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DEGRADATION AND CHARACTERIZATION OF ANTIMISTING KEROSENE (AMK)

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INTERIM REPORT

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16. Abstract

The effect of elongational flow on polymer degradation has been studied by forcing AMK through metal screens and packed tubes at high velocities. Effects of screen size, bead size, and tube length on degrader power have been determined. At a specific power of 15 kWs/l (30 HP at 10,000 lb/hr), AMK exhibits filtration and ignition properties similar to Jet A in small-scale tests.

A glycol/amine carrier fluid that was originally developed to promote rapid dissolution of FM-9 polymer in Jet A has been found to increase antimisting effectiveness, reduce gel formation and filtration resistance, and require less degrader power. Other fuel-soluble hydrogen bonding agents have been found to produce similar effects with FM-9 in Jet A.

At low Reynolds numbers, the flow of AMK through metal screens and paper filters is characterized by a critical velocity that depends on polymer degradation, filter material, pore size, and presence of hydrogen bonding agents. Below this critical velocity, the flow resistance of AMK is determined by the low shear viscosity which is only 1.6 times higher than Jet A. At a slightly higher velocity, the flow resistance increases dramatically. While this phenomenon is commonly observed with many polymer solutions, in the case of FM-9 it is also associated with gel formation that may result in filter plugging. However, at very high velocities, gel formation and filter plugging are no longer evident with either metal screens or packed tubes.

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LIST OF SYMBOLS AND ABBREVIATIONS

A	area of filter
AMK	antimisting kerosene
ASTM	American Society of Testing and Materials
cp	centipoise
cSt	centistokes
D	diameter
dp	bead size
FAA	Federal Aviation Administration
FR	filtration ratio (filtration velocity of Jet A relative to AMK)
(FR)i	initial filtration ratio (undegraded AMK)
HP	horsepower
ICI	Imperial Chemical Industries
ID	inner diameter
JPL	Jet Propulsion Laboratory
k	permeability
kWs/l	kilowatts•sec/liter
L	length of tube
LIR	Lower Ignition Resistance
Q	flow rate
RAE	Royal Aircraft Establishment
R_e	Reynolds number through porous media (Vdp/η)
SwRI	Southwest Research Institute
UIR	Upper Ignition Resistance
$V = Q/A$	superficial velocity
VR	viscosity ratio of AMK relative to Jet A
ΔP	pressure drop
μ	shear viscosity
η	kinematic viscosity

I. INTRODUCTION

Antimisting kerosene (AMK) fuel contains a high molecular weight polymer (FM-9)* that resists the formation of small droplets and has been shown to improve fire safety in simulated tests of ground crash conditions[1,2]**; however, poor fuel filtration and atomization characteristics preclude its use in aircraft turbine engines and fuel systems until acceptable means of restoring these essential fuel properties can be developed.

While both shear and elongational effects can contribute to polymer degradation, it is expected that elongational flows will be more destructive than shear flows.[3] Some insight for this expectation can be provided by considering the differences between these two types of flows. The familiar shear flow that is commonly produced in a long capillary tube is characterized by a velocity gradient that is normal to the direction flow. As a dilute polymer solution flows through the capillary, polymer molecules tend to be stretched along the principal axis of stress. However, shear flows are rotational; therefore, polymer molecules rotate at a frequency of one-half the shear rate.[4] Because of this rotational motion, polymer molecules cannot stay oriented in the stress field; consequently, very little polymer deformation occurs with a low viscosity solvent even at high rates of shear.[3] Elongational flows occur in many practical situations, such as in the entrance region to a capillary tube, in flow through an orifice, and in porous media. In all of these examples, the velocity gradient is in the direction of flow. Since this type of flow is irrotational, a polymer molecule would be expected to stay oriented in the stress field longer; therefore, larger polymer deformations should occur.

Because of the presence of solid boundaries, most flows are a combination of both shear and elongation; however, by an appropriate choice of geometry, one type of flow can be made to dominate. For example, laminar flow in a long capillary tube is primarily a shear flow except for the entrance region. On the other hand, in a very short tube ($L/D<1$) or for a tube filled with beads, the flow is predominantly elongational.[5]

Since elongational flow is expected to be more effective in producing polymer degradation than shear flow, the primary objective of this work is to determine the effect of the sudden acceleration produced by flow into the pores of metal screens or tubes packed with beads on the mist ignition and filtration properties of AMK. However, relatively few standard tests have been found to be useful in measuring the performance characteristics of AMK; therefore, a second but equally important part of this work is the development of test methods that relate to mist flammability and rheological properties of AMK.

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* This is a proprietary polymer that was provided by ICI Americas, Inc., Wilmington DE 19897.

** Underscored numbers in parentheses refer to the list of references at the end of this report.

II. EXPERIMENTAL PROCEDURES

Polymer degradation experiments were conducted by displacing AMK from a large bore (12.7 cm) hydraulic cylinder. Before each run, approximately 6 liters of AMK were charged to the rod side of the cylinder. A high-pressure variable flow pump forced hydraulic fluid into the piston side of the cylinder, thus displacing AMK and forcing it to flow through the metal screens or packed tubes. The volumetric flow rate (Q) was the independent variable and was generally increased from a low value to the highest value (this was limited either by the maximum pressure of 4000 psi or by the maximum flow rate of 5 gal/min) and then back to an intermediate flow rate. This procedure was used to help detect hysteresis effects that might occur due to filter plugging. The pressure drop (ΔP) was measured with a gauge and transducer, with the output of the latter recorded as a function of flow time.

Metal screens* were held in place by two metal discs (1/2 x 1/16 inch) that fit into a 1/2-inch union. The exposed area of the screens were determined by the size and number of holes in the discs. Stainless steel tubes (1/4 inch) were packed with uniform glass beads.** A 1/4-inch union was drilled out to allow it to be fitted with a 1/4-inch tube (0.2 - 1.9 cm in length). Metal screens (100 mesh) were placed at both ends of the tube to retain the beads, and a backup plate was used to support the screen on the downstream side. Polymer degradation was measured in terms of changes in the viscosity ratio (VR) and the filtration ratio (FR) of AMK relative to Jet A, and mist flammability.

At the beginning of this program, it was agreed by the US/UK Technical Committee that a standardized filtration test would be the primary measure of intentional degradation. Basically, this test utilizes a viscometer in which a specific volume (96 ml) of fuel flows between two timing marks on a vertically mounted glass tube (2.5 cm ID). A 16- to 18-micrometer Dutch weave screen is attached to the bottom of the tube and the flow time for AMK relative to Jet A is reported as the FR. For undegraded AMK at 25°C, this ratio is generally between 40 and 50; however, degradation can reduce this ratio to close to 1.0.

More detailed experiments were conducted by maintaining a fixed gas pressure over a fluid reservoir and collecting a quantity of filtrate over a timed interval. This procedure is similar to the standard test, but it has several important advantages. In the first place, by measuring the flow rate or velocity at different pressures, more information can be obtained on the rheological behavior of AMK. In particular, it can be seen that AMK can flow through a filter without gel formation and without abnormal resistance if the superficial velocity ($V = Q/A$, where A is the filter area) is less than a critical value. Also, by making measurements at increasing and decreasing pressures, effects of filter plugging can be easily detected.

* M-8656 (nominal 40 microns) and M-7211 (nominal 10 microns), stainless steel, Dutch weave, Purolator Inc., Newbury Park, California.

** Ferro Microbeads, Jackson, Mississippi

Last and most important, this test procedure allows experiments to be conducted for longer times than the standard filtration test. Flow time is a particularly critical factor with highly degraded AMK.

In order to more closely simulate filtration conditions in an aircraft fuel delivery system, a filtration test was devised in which a gear pump forces AMK through different types of filters at a specified flow rate. The pressure drop across the filter was measured for 2 minutes with a transducer and recorder.

The mist flammability of AMK was measured with a spinning disc atomizer that has been described in an earlier report.(6) In order to make these experiments less subjective, a radiometer and recorder were incorporated to measure the growth of flame propagation as a function of the tangential velocity of the disc. Standardized tests were used to measure the kinematic viscosity (ASTM D-445), the polymer content (ASTM D-381), and orifice flow cup characteristics (British Standard 1733).

III. EXPERIMENTAL RESULTS

A. Effect of Polar Additives on Mist Flammability of FM-9 in Jet A

In order to rapidly dissolve the FM-9 polymer in Jet A, a carrier fluid consisting of a fuel-soluble glycol and amine was developed by ICI Ltd. Early in this program, it was reported that AMK consisting of FM-9 and carrier fluid appeared to have a higher resistance to mist ignition than FM-9 in Jet A without the carrier fluid.(6) It was also reported that fuel-soluble surfactants that eliminated gel formation also increased the antimisting effectiveness of FM-9.(6) These conclusions were based on experiments with the spinning disc before the radiometer had been adopted to measure flame radiation. In these earlier experiments, the mist flammability of a fuel was expressed in terms of the lower ignition resistance (LIR) and the upper ignition resistance (UIR). The former was determined by bracketing the lowest speed at which ignition and flame propagation began, while the latter was obtained by bracketing the disc speed at which the disc was engulfed in flames. Because of the subjective nature of these flammability criteria, experiments were conducted to determine the effect of carrier fluid and surfactants on the mist flammability of FM-9 in Jet A as sensed by a radiometer. The results in Figure 1 show the effect of disc velocity on flame radiation for the same fuel flow rate (1.0 l/min) that was used in the early experiments. The flame radiation of Jet A increases rapidly at velocities above 15 m/s; with 0.3% FM-9 the flame radiation is negligible up to 58 m/s. The improved antimisting effectiveness of FM-9 with carrier fluid is evident by the lower radiation level and the higher critical disc velocity (i.e., $V_c \cong 83$ m/s). The effect of adding the surfactant (SO-A)* is also clearly evident but the critical velocity is not well defined. It is particularly important to note that FM-9 made with glycol produced no measurable flame radiation at velocities up to 95 m/s.

The effects of increasing the fuel flow rate to 2.0 and 2.8 l/min are shown in Figures 2 and 3, respectively. There appears to be a slight reduction in the critical velocity at 2.0 compared to 1.0 l/min; however, the primary effect of the higher fuel flow rate is to increase the total radiation and to make the onset of flame propagation more distinctive. Increasing the flow to 2.8 l/min (which was the maximum output of the fuel pump) produced no significant change in the critical velocity; consequently, a standard fuel flow rate of 2.0 l/min was used in all subsequent flammability experiments.

It is evident that the results of these recent experiments are in good agreement with those reported earlier regarding the effect of the carrier fluid. However, spinning disc experiments have also shown that Jet A containing

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* Scher Chemicals, Inc., Industrial West, Clifton, NJ 07012.

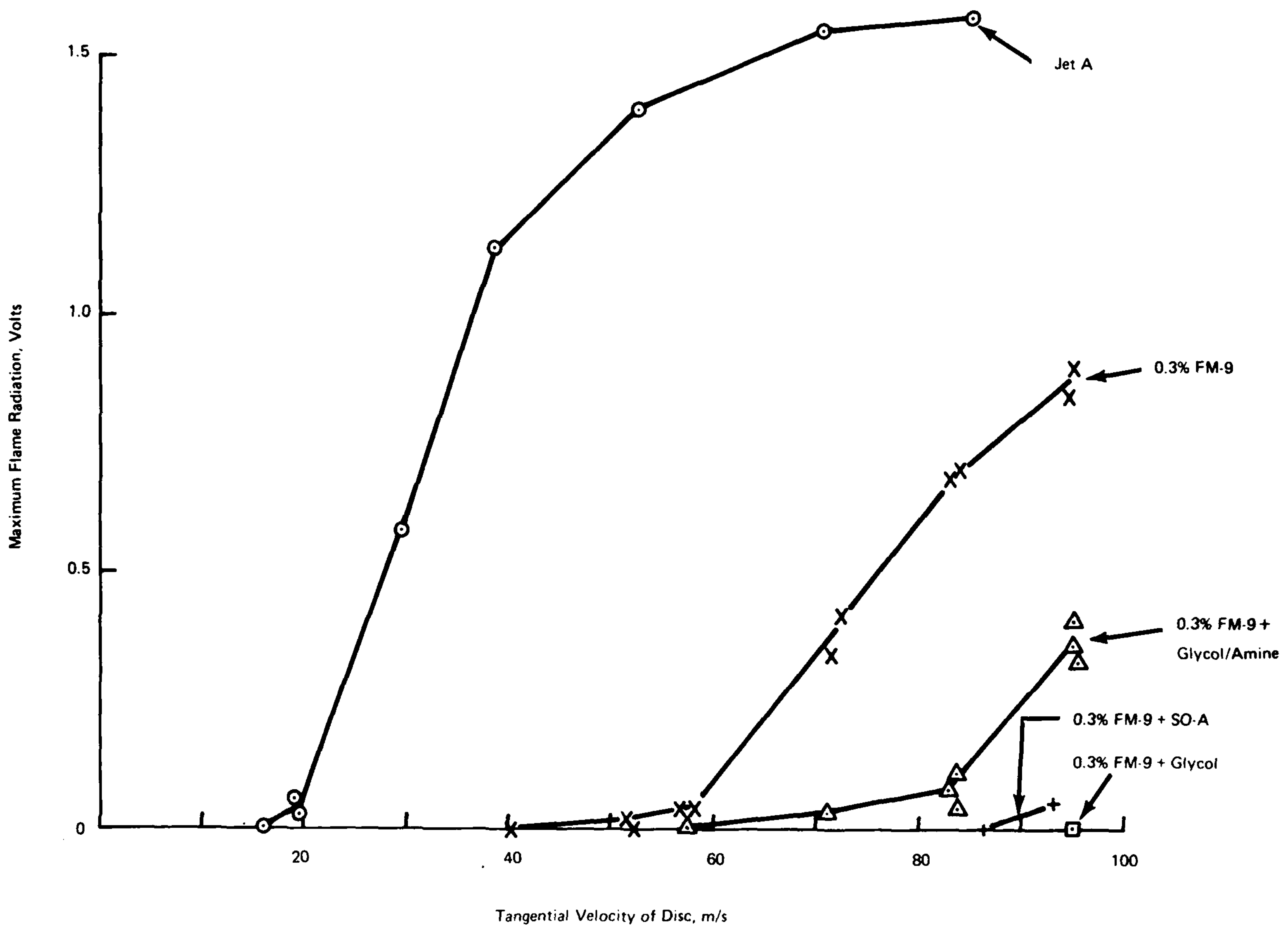

FIGURE 1. EFFECT OF CARRIER FLUID COMPONENTS AND SO-A ON MIST FLAMMABILITY OF FM-9 IN JET A - FUEL FLOW = 1.0 ℓ/min.

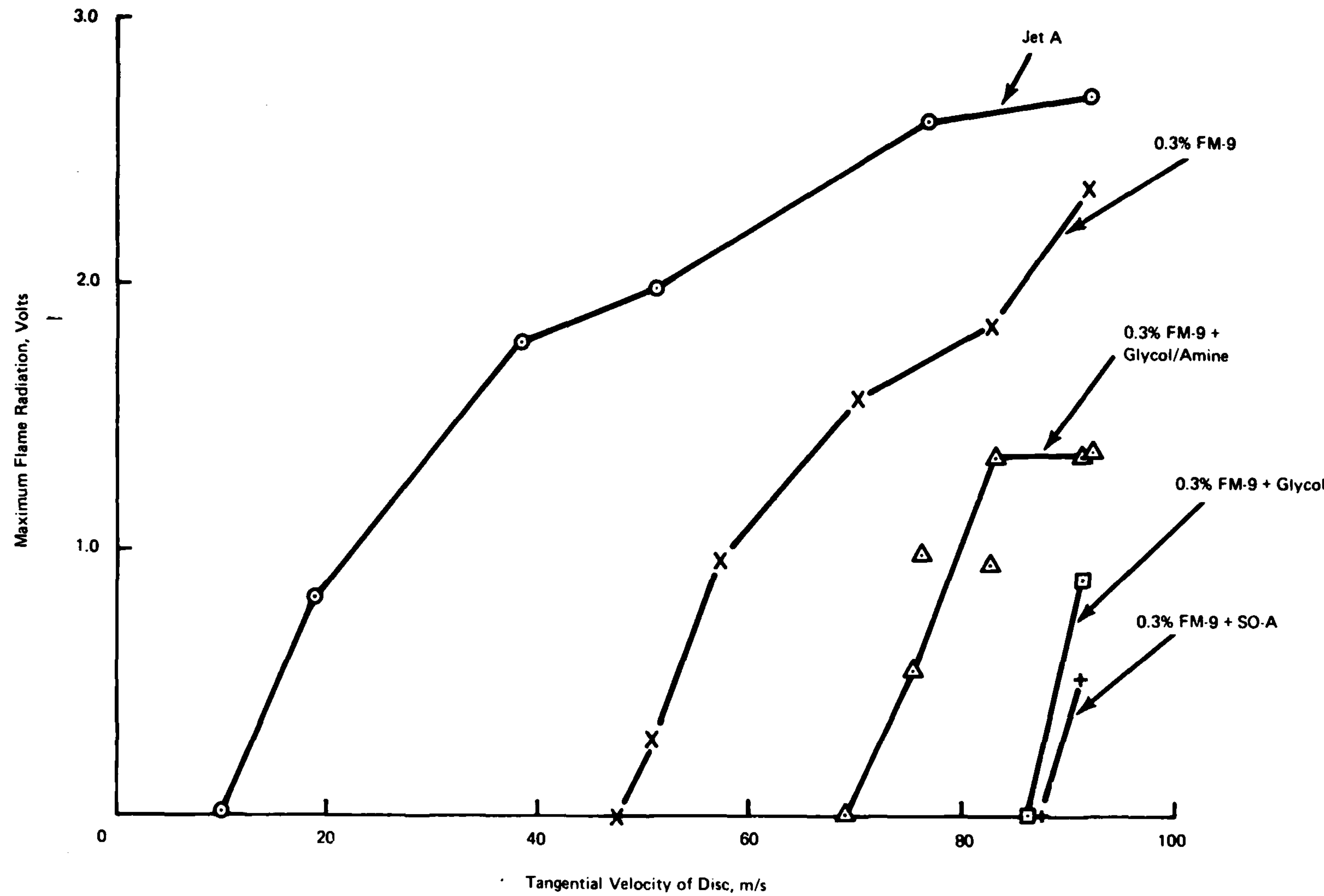

FIGURE 2. EFFECT OF CARRIER FLUID COMPONENTS AND SO-A ON MIST FLAMMABILITY OF FM-9 IN JET A - FUEL FLOW = 2.0 ℓ/min.

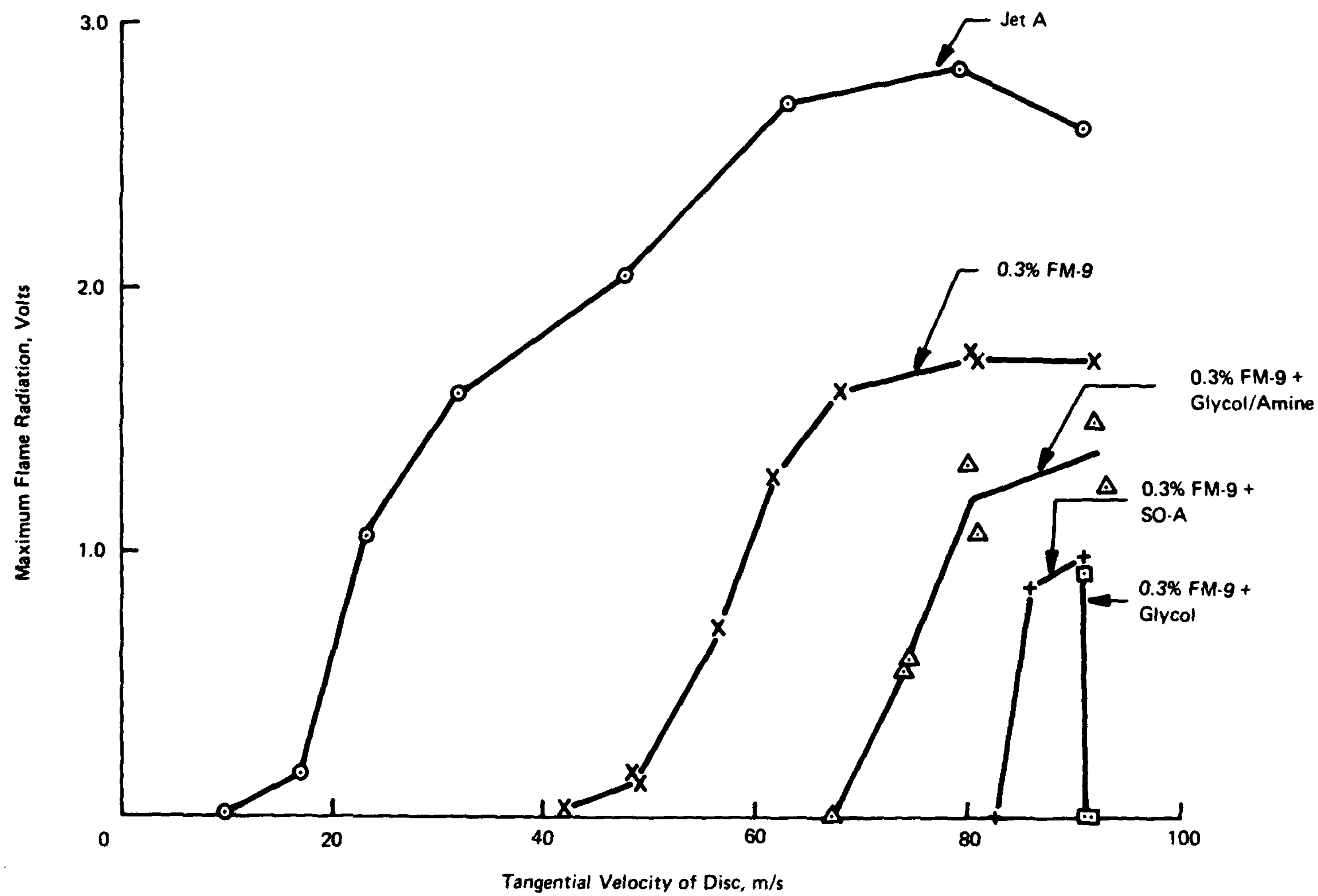

FIGURE 3. EFFECT OF CARRIER FLUID COMPONENTS AND SO-A ON MIST FLAMMABILITY OF FM-9 IN JET A - FUEL FLOW = 2.8 ℓ/min.

FM-9 and only the glycol component of the carrier fluid (i.e., no amine) has a slightly higher critical velocity than standard AMK blends. These findings are in conflict with rocket-sled results in which AMK is reportedly unable to pass the standard two-rocket test (66 m/s) unless it contains both carrier fluid components.* While it is possible that this difference is a characteristic related to small-scale fire tests, the spinning disc test has reliably predicted several aspects of AMK behavior that have closely corresponded with large-scale fuel spillage tests. In particular, it has shown that the carrier fluid improves the fire resistance of FM-9 in Jet A. More specifically, the critical velocity increases from approximately 50 m/s for 0.3% FM-9 without carrier fluid (pass/marginal condition for China Lake tests)[(1)] to approximately 70 m/s for 0.3% FM-9 with carrier fluid (pass/marginal conditions for FAA Technical Center** tests).[(2)] This good agreement between the critical disc velocity and the pass/marginal air velocity is also evident for different concentrations of FM-9 (Figure 4). Because of the need for a reliable small-scale flammability test, it is important to determine what aspect of the spinning disc test could be responsible for these different results with respect to the importance of the amine component of the carrier fluid. Consequently, the technical committee decided that Jet Propulsion Laboratory (JPL) would conduct intermediate-scale fire tests. Samples of these two fuels (i.e., standard AMK and AMK without amine) were sent to JPL. It was reported that FM-9 without amine failed the JPL test at air velocities as low as 45 m/s, while the standard AMK passed at air velocities exceeding 80 m/s.*** To determine how the JPL test differed from the spinning disc test, JPL agreed to retest the fuels. Additional samples of fuel with the following physical and chemical properties were sent t JPL:

% FM-9	Glycol	Amine	VR	FR	Orifice Flow	Critical Disc Velocity, m/s
0.305	yes	yes	1.69	52	2.6	65
0.307	yes	no	1.89	14	7.0	74

Again the JPL test rated the sample with both carrier fluid components a pass at air velocities up to 80 m/s and the sample without the amine a fail at 50 m/s. However, the pass/fail criterion in the JPL test, which is taken as a flame length of 1 meter, appeared to be different from the critical velocity in the spinning disc test. For example, while the standard AMK

* Verbal comments by Dr. S.P. Wilford, Royal Aircraft Establishment (RAE), 5th Joint Technical Committee Meeting on Antimisting Fuel, November 1979.

** Federal Aviation Administration Technical Center.

***Verbal communication with Dr. V. Sarohia, JPL, December 1979.

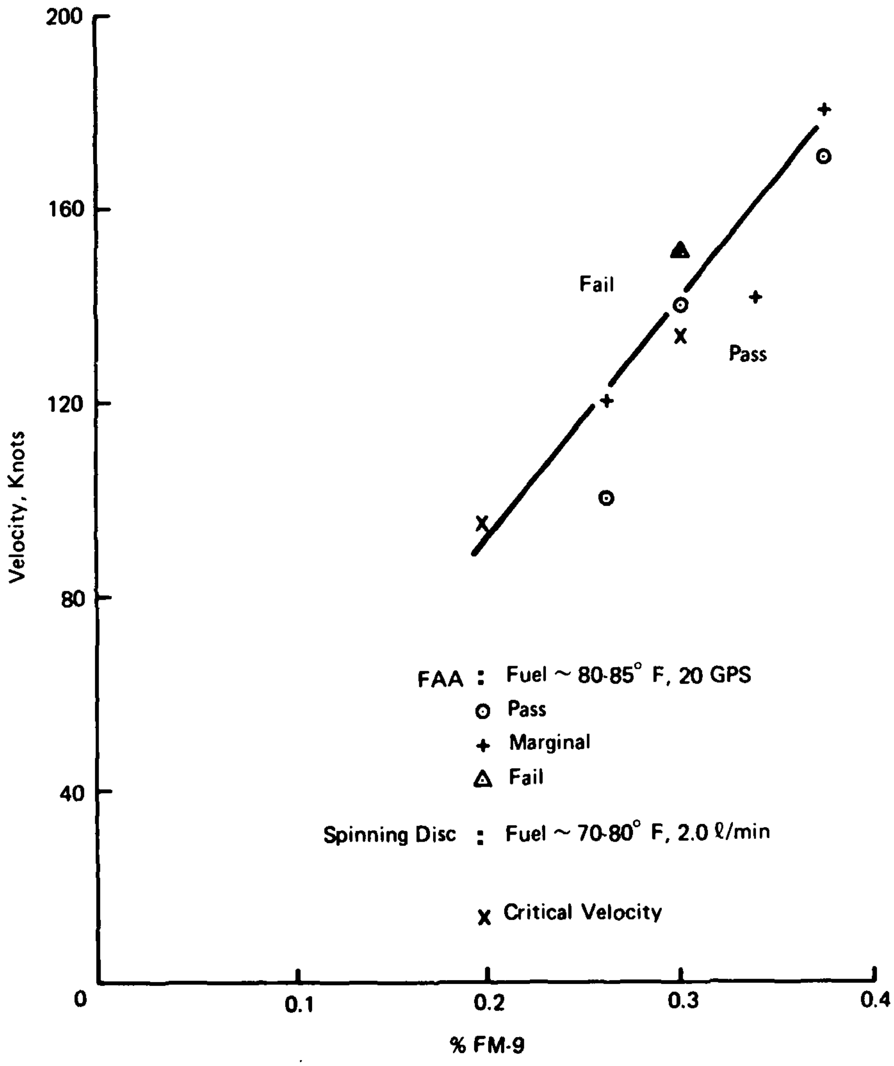

FIGURE 4. COMPARISON OF SPINNING DISC CRITICAL VELOCITY WITH PASS/MARGINAL VELOCITY IN FUEL SPILLAGE TEST

passed at velocities up to 80 m/s, the flame length in all these cases appeared to be about 1/2 to 3/4 meter and did not increase significantly with air velocity from 50-80 m/s. More importantly, the flame length for the sample without amine, which was rated as a fail at 50 m/s (i.e., had a flame length of approximately 1 m), did not increase significantly when the air velocity was increased to 80 m/s. These results are very different from the critical velocity in the spinning disc test. In this test, the degree of flame propagation (as measured by a radiometer) increases rapidly when the critical velocity is exceeded by as little as 5 to 10 m/s.

Other fuel samples were tested that were blended by ICI Americas, Inc. While standard AMK samples blended by Southwest Research Institute (SwRI) and ICI appeared to produce the same flame length, three other samples that contained only the glycol component of the carrier fluid produced different results (two appeared worse than the standard AMK and one appeared better). Since there is some doubt about the uniformity of the ICI blends made without amine, the slightly inferior performance reported for some of these blends in the JPL test should not be considered conclusive proof that the amine is necessary for adequate mist flammability protection.[7]

The reason for good agreement between standard AMK blends made by ICI and SwRI but poor agreement for different ICI blends without amine is not presently understood. However, several different samples of standard AMK, both those blended by ICI and SwRI, have been found to be characterized by approximately the same critical velocity (66-71 m/s). On the other hand, while most blends made without amine fall in this same range, others have been found that are significantly lower (55 m/s) and also higher (85 m/s). The reason for these differences was not evident in any of the physical properties such as orifice flow, filtration ratio, or viscosity ratio. Therefore, it is imperative that future attempts to compare the results of different flammability tests should have a sufficient quantity of fuel prepared so that all the participants can obtain a representative sample.

B. Effect of Temperature on Mist Flammability of FM-9 in Jet A

Because of the potential importance of temperature on mist flammability, spinning disc experiments were conducted with heated AMK. In a typical mist ignition test with fuel at ambient temperature, the disc speed is set and then the fuel pump is started. Fuel flows through approximately 21 feet of 1/4-inch insulated tubing before it enters the central cavity of the spinning disc. After 3 seconds, the fuel flow is stopped and the disc speed changed to the next test condition. While this procedure is satisfactory at ambient fuel temperatures, it does not work well at elevated temperatures. For example, the temperature of the fuel in the insulated line decreases as soon as flow is stopped and increases as soon as the flow is restarted. To minimize these effects, the fuel on-time was increased and the fuel off-time decreased in experiments with heated fuel. Although this approach was partially effective, the fuel temperature was not constant during these tests and typically increased 2° to 3°C. Fuel temperatures were measured

by an iron-constantine thermocouple (wire diameter~0.01 inch) and digital readout that was accurate to 0.5°C. The thermocouple was located at the discharge end of the fuel line. It was felt that little temperature change would occur due to heat transfer to the disc. However, prior to the ignition experiments, a second thermocouple was placed in the fuel stream that issued from the disc. Since no measurable difference could be detected between these two thermocouples, it is reasonable to conclude that the thermocouple in the line estimated the temperature of the fuel just before contact with the ignition source at least to within 0.5°C.

Although the spinning disc test in its current configuration is not ideally suited to investigate fuel temperature effects with a high degree of precision, the results presented in Figure 5 indicate that fuel temperatures in the range of 20° to 40°C (flash point = 51°C) have little effect on either the critical ignition velocity or the degree of flame propagation following ignition. These findings appear to be in agreement with rocket-sled results which showed little or no temperature dependency over approximately the same range.[8] However, they do not agree with the JPL fire test results which indicate a substantial decrease in the pass/fail envelope over a relatively small change in fuel temperatures; i.e., 80 to 90 m/s at 21°C and 50 to 70 m/s at 32°C.[9]

C. Degradation of AMK With Metal Screens

The data in Figure 6 are typical of the flow characteristics of Jet A and AMK through metal screens at high Reynolds numbers ($R_e = dp\ V/\eta$, where dp is a characteristic pore size, V = Q/A is the superficial velocity, and η is the kinematic viscosity). Since the pressure drop (ΔP) across the filters is a measure of specific degrader power, it has been expressed in terms of kWs/l instead of the more conventional units of force per unit area. The data for Jet A (1.9 cSt) in the 40-micrometer screen is representative of $100<R_e<1000$. Since inertial effects become important in porous media for $R_e \gg 1$, the nonlinear increase in flow resistance with velocity in Figure 6 is due primarily to inertial rather than viscous effects. Despite the fact that the flow of AMK through porous media (metal screens, packed tubes and paper filters, etc.) is characterized by gel formation and filter plugging at low velocities (see Section E), the flow resistance of AMK at 25°C was essentially the same as Jet A at high velocities. The absence of filter plugging is emphasized by the typical pressure-time traces (Figure 7) in which the pressure drop across the filter did not increase significantly with time. These results are quite surprising and suggest that a high superficial velocity through the screen may prevent gel formation. While the reason for this behavior is not well understood, it may be that the induction time for gel formation is longer than the flow time. Nevertheless, these results suggest that specially designed screens which maintain a high superficial velocity may be able to filter AMK without the gel formation that is experienced at low velocities.

While the flow of AMK in the 40-micrometer screen was essentially identical to Jet A, quite different results were observed for the 10-micrometer screen. In this case, the resistance of AMK was higher than Jet A at low velocities

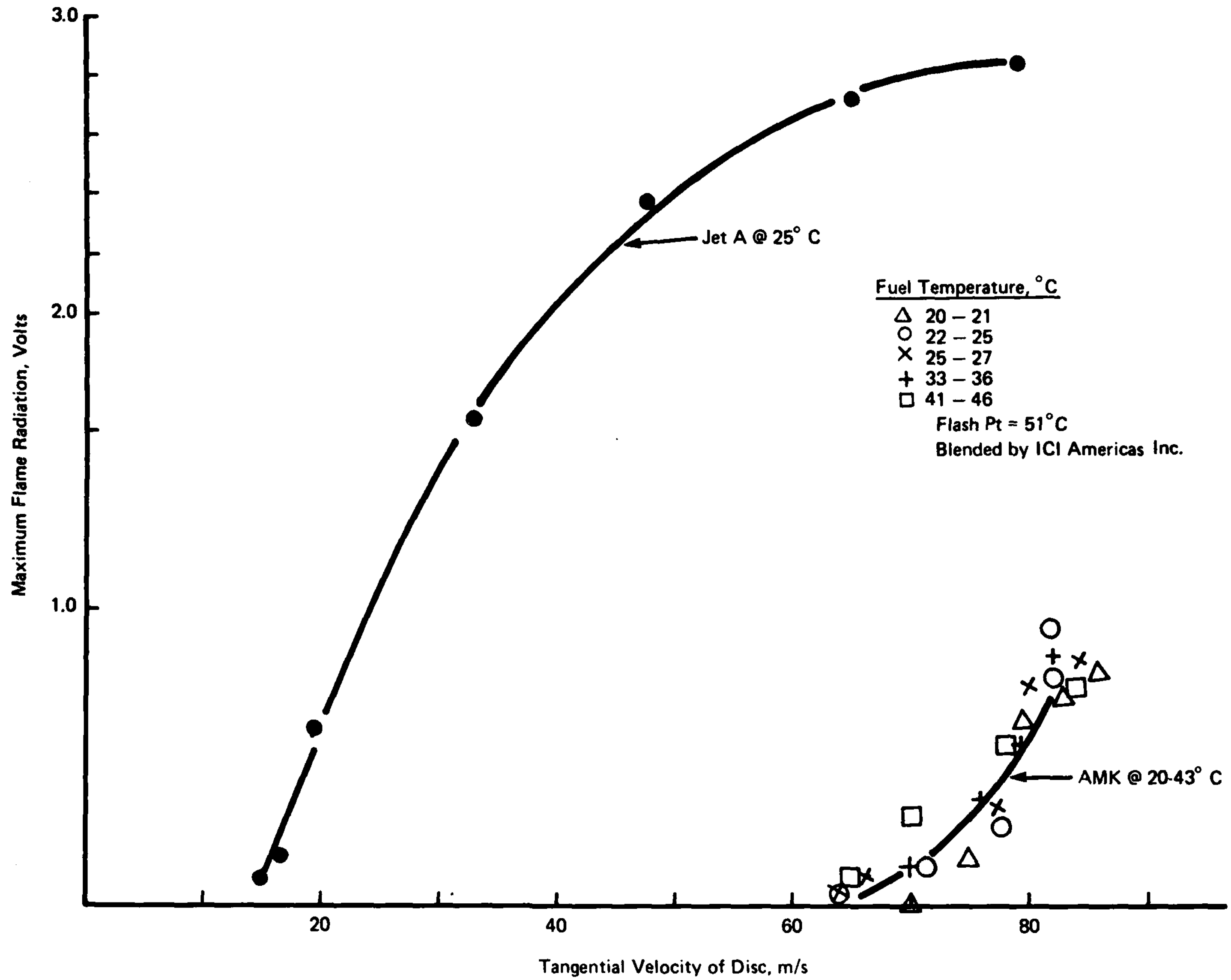

FIGURE 5. EFFECT OF TEMPERATURE ON MIST FLAMMABILITY OF AMK

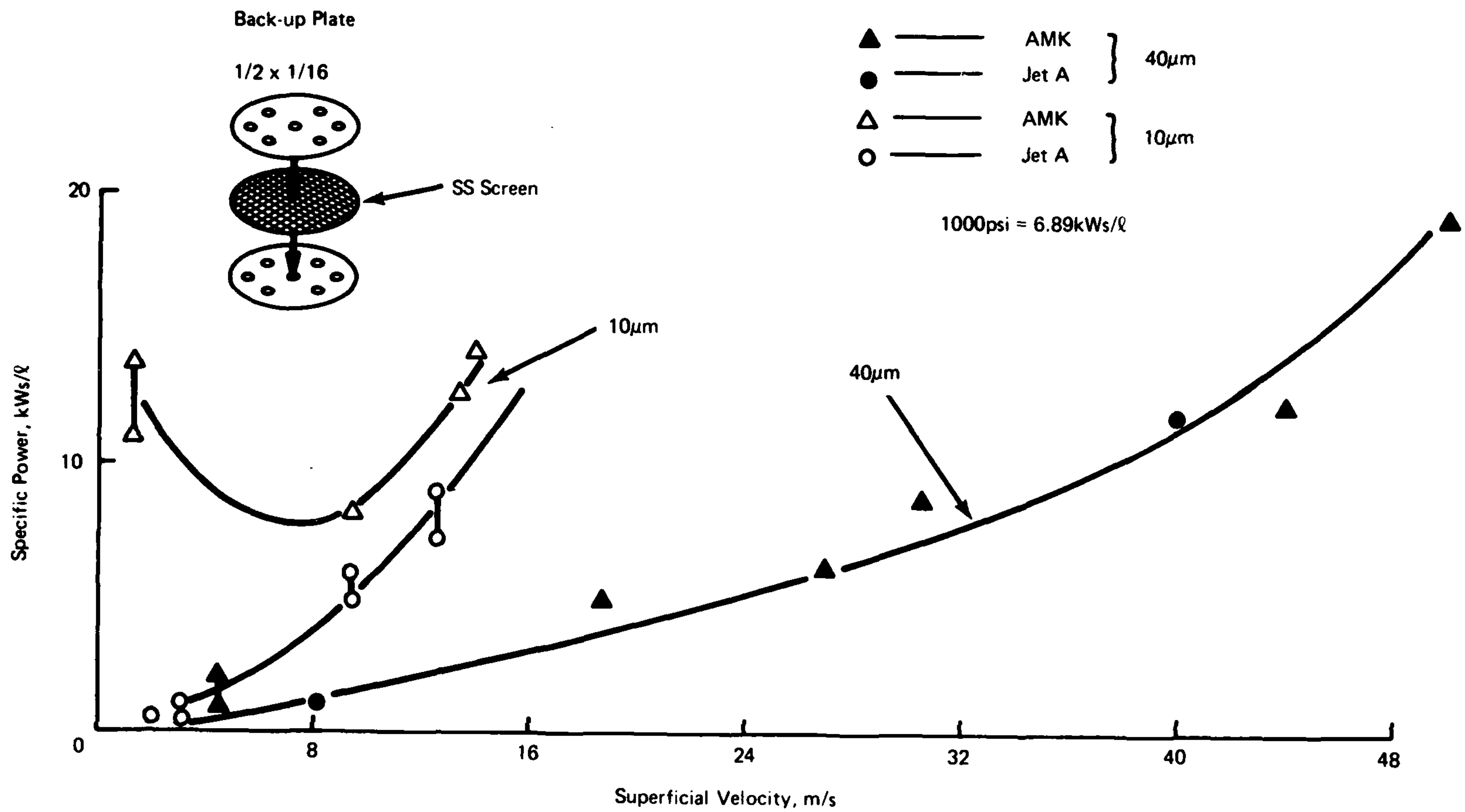

FIGURE 6. FLOW OF AMK THROUGH METAL SCREENS

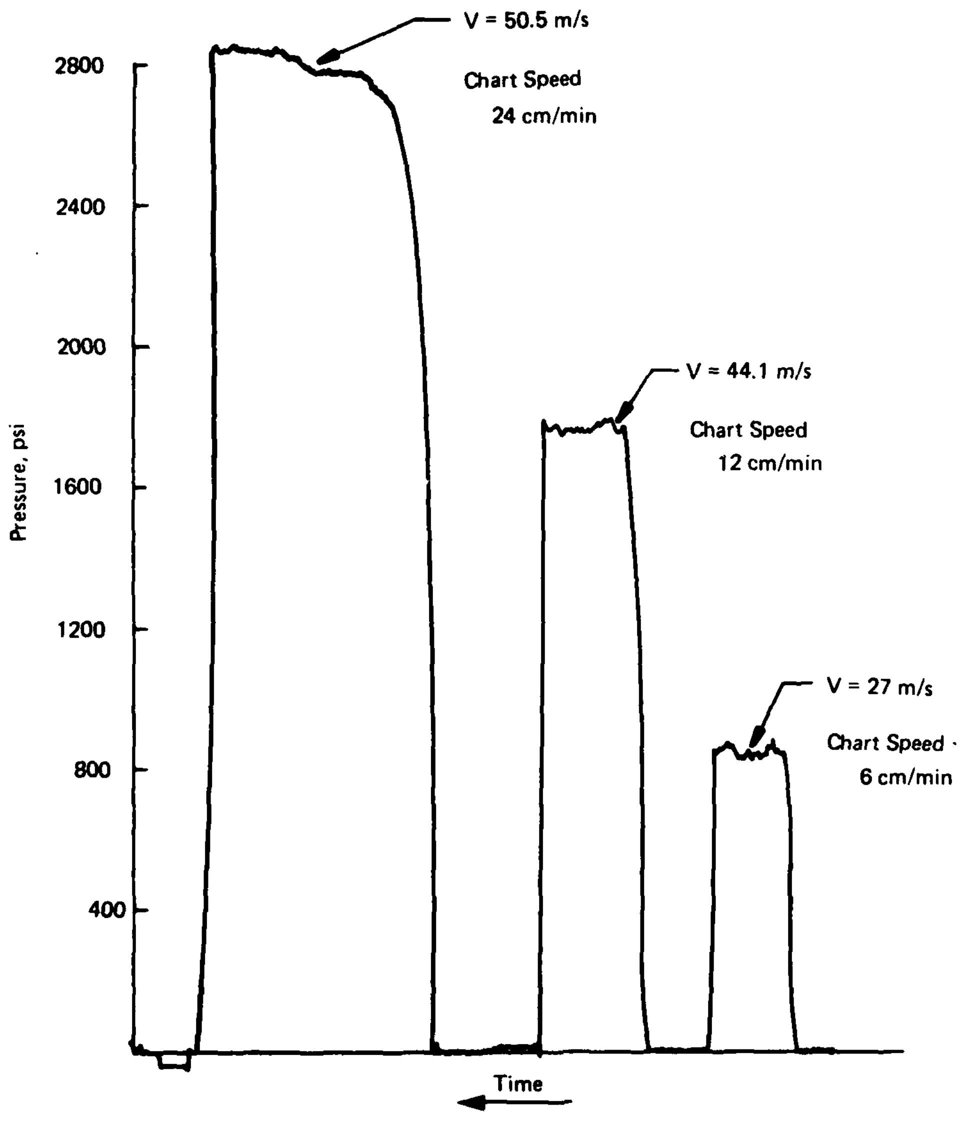

FIGURE 7. PRESSURE-TIME TRACE FOR FLOW OF AMK THROUGH 40-μm SCREEN (A = 0.045 cm^2)

but approached Jet A as the velocity increased! This unusual behavior in which the pressure is not uniquely determined by the velocity is also demonstrated by the pressure-time traces for AMK shown in Figure 8. For example, at 1.2 and 13.4 m/s, the pressure drop is almost the same value. Although there is a possible trend of increasing pressure with time for the low-velocity data, the effect is small over the relatively short time scale of these experiments (1.5 minutes). It is suspected that this anomaly is associated with gel formation and filter plugging; consequently, degradation results for the 10-micrometer screen should be viewed with caution until longer term experiments can be conducted. Nevertheless, it is important to note that at high velocities, the flow resistance of AMK approached that of Jet A even with the 10-micrometer screen.

The effect of superficial velocity on the level of degradation (as measured by the viscosity ratio) for flow through the metal screens is shown in Figure 9. In the case of the 40-micrometer screen, the viscosity ratio decreased with increasing velocity. It is important to point out that the theory of intrinsic viscosity relates the reduction in the viscosity ratio to a reduction in the molecular size of a polymer molecule[10]. The anomalous flow of AMK through the 10-micrometer screen that was previously discussed is again evident in Figure 9. In this case, the viscosity ratio appears to be essentially independent of the velocity.

The results in Figure 10 show the effect of specific degrader power (ΔP) on the change in the viscosity ratio of AMK produced by flow through 40- and 10-micrometer metal screens. Both screens produced the same decreasing viscosity ratio with increasing degrader power, and there was no significant difference in the power required to produce a particular level of degradation. The results in Figure 11 show that the filtration ratio is more sensitive to the screen size than the viscosity ratio. This may be due in part to the relatively poor experimental repeatability that is sometimes experienced in measuring the filtration ratio. It has recently been discovered that if the amine component of the carrier fluid is added before the polymer is fully in solution, the resulting blend will have a high and sometimes very erratic filtration ratio. Since the filtration ratio of this undegraded AMK sample was higher than normal (65 compared to 45), the poor experimental repeatability for the filtration ratios in Fig. 11 is probably the result of improper blending. In any event, these results indicate that the resistance of AMK to flow through a filter can be virtually eliminated. If it can be established that screens can be used continuously for prolonged periods of time, this high velocity filter-degrader may provide an alternate approach to conventional aircraft filters.

D. Degradation in Packed Tubes

The flow curves for AMK in tubes packed with 840-micrometer glass beads (Figure 12) were almost identical to those of Jet A and are representative of the high R_e flow regime in which the pressure drop increases with the superficial velocity in a nonlinear manner. Figure 13 shows the effect of increasing the dimensionless tube length (L/dp) on the viscosity ratio of AMK. At a given velocity, a higher degree of degradation (lower viscosity ratio) can be produced by increasing the tube length. On the other hand, the results in Figure 14 show that the increased degradation in the longer tubes is counterbalanced by the requirement of a higher specific degrader power.

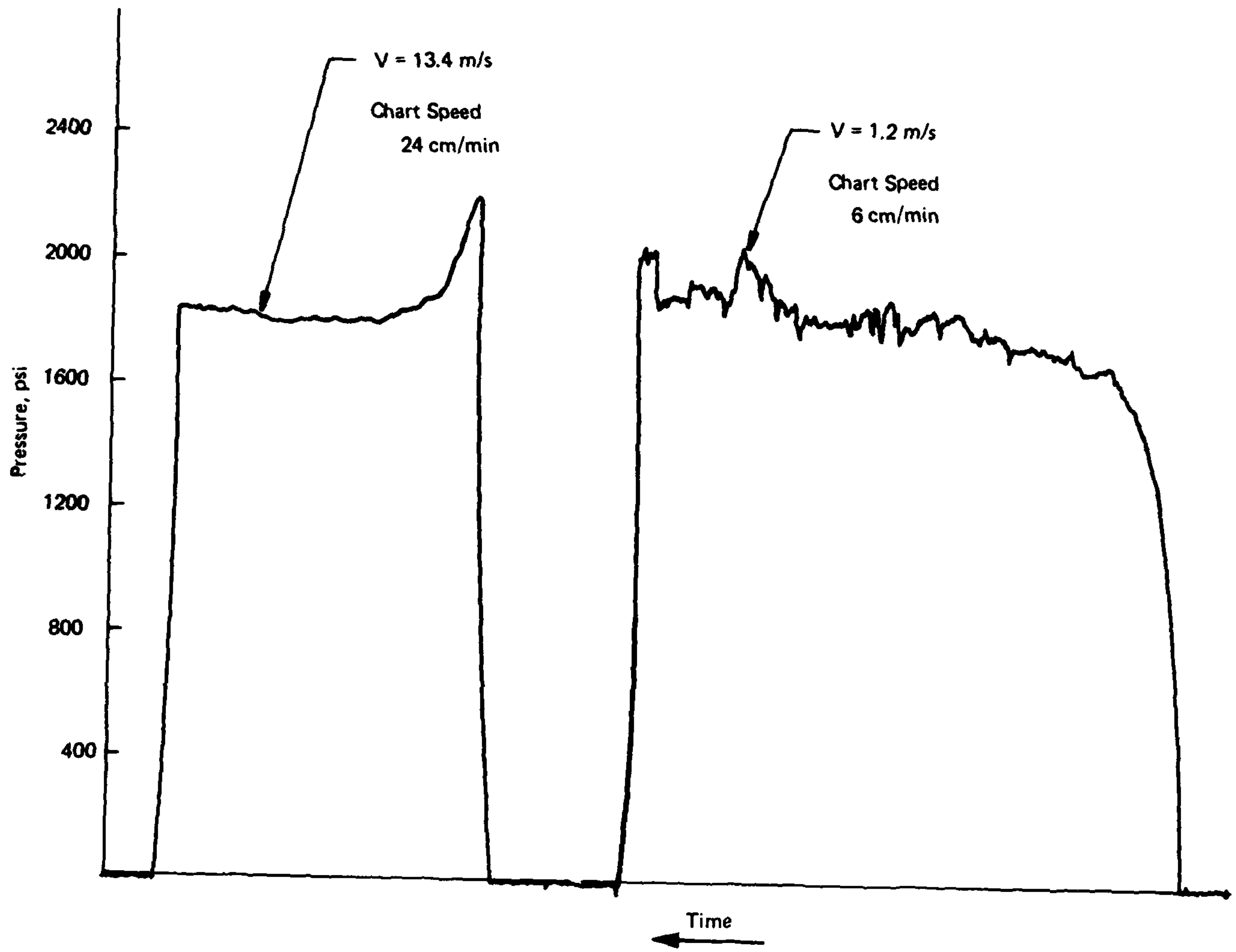

FIGURE 8. PRESSURE-TIME TRACE FOR FLOW OF AMK THROUGH 10-μm SCREEN (A = 0.184 cm^2)

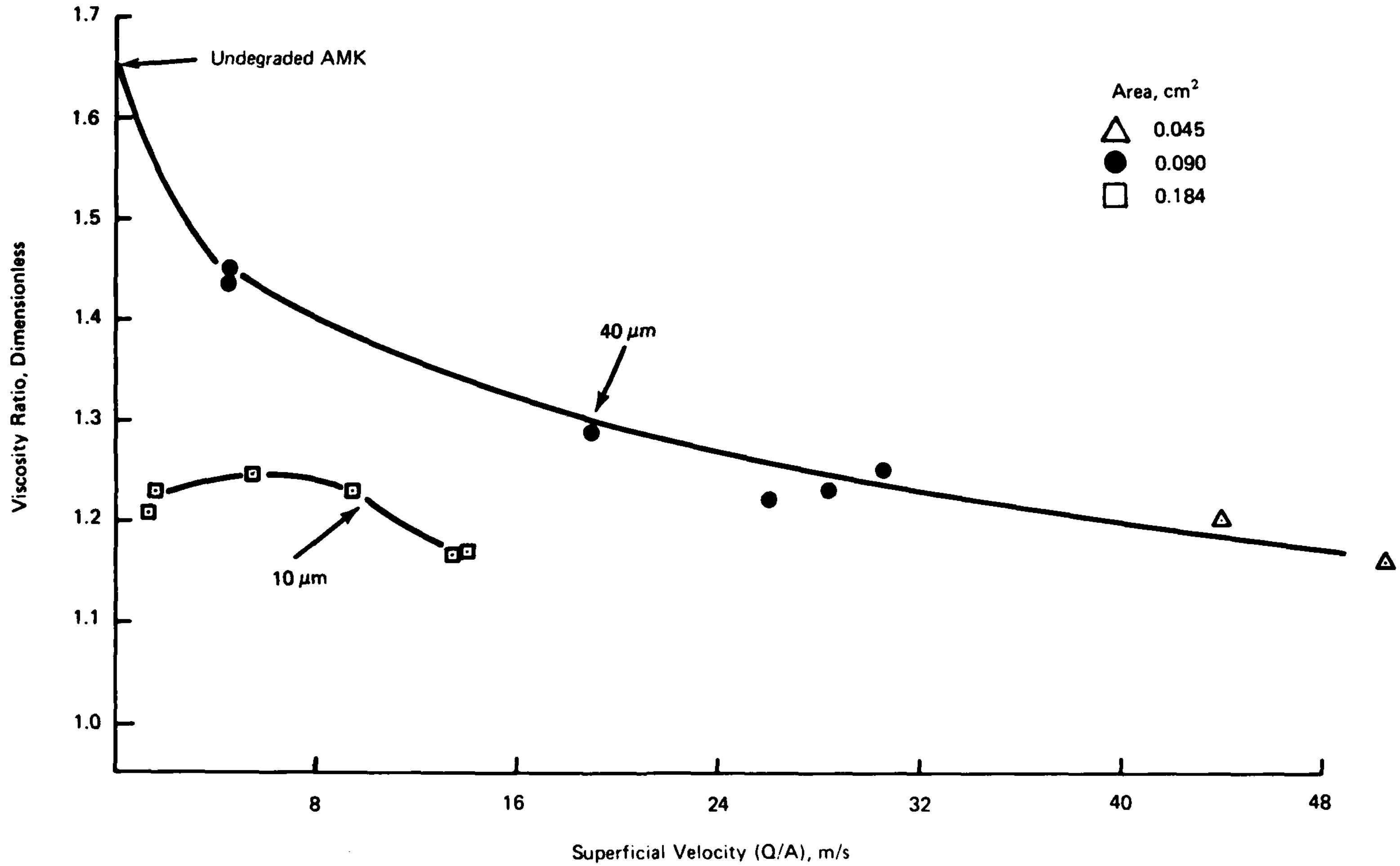

FIGURE 9. EFFECT OF SUPERFICIAL VELOCITY THROUGH METAL SCREENS ON THE VISCOSITY RATIO

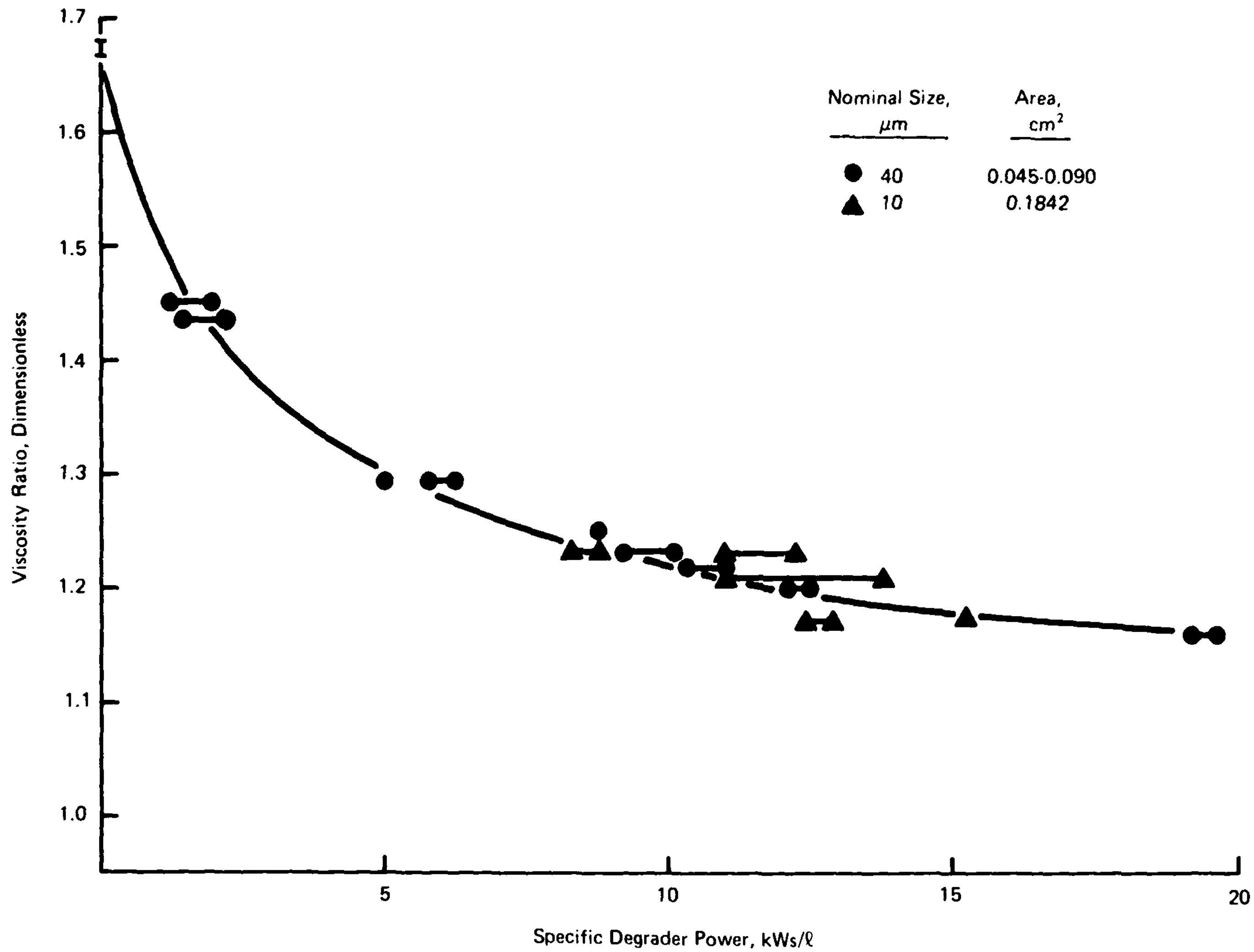

FIGURE 10. EFFECT OF SPECIFIC DEGRADER POWER THROUGH METAL SCREENS ON THE VISCOSITY RATIO

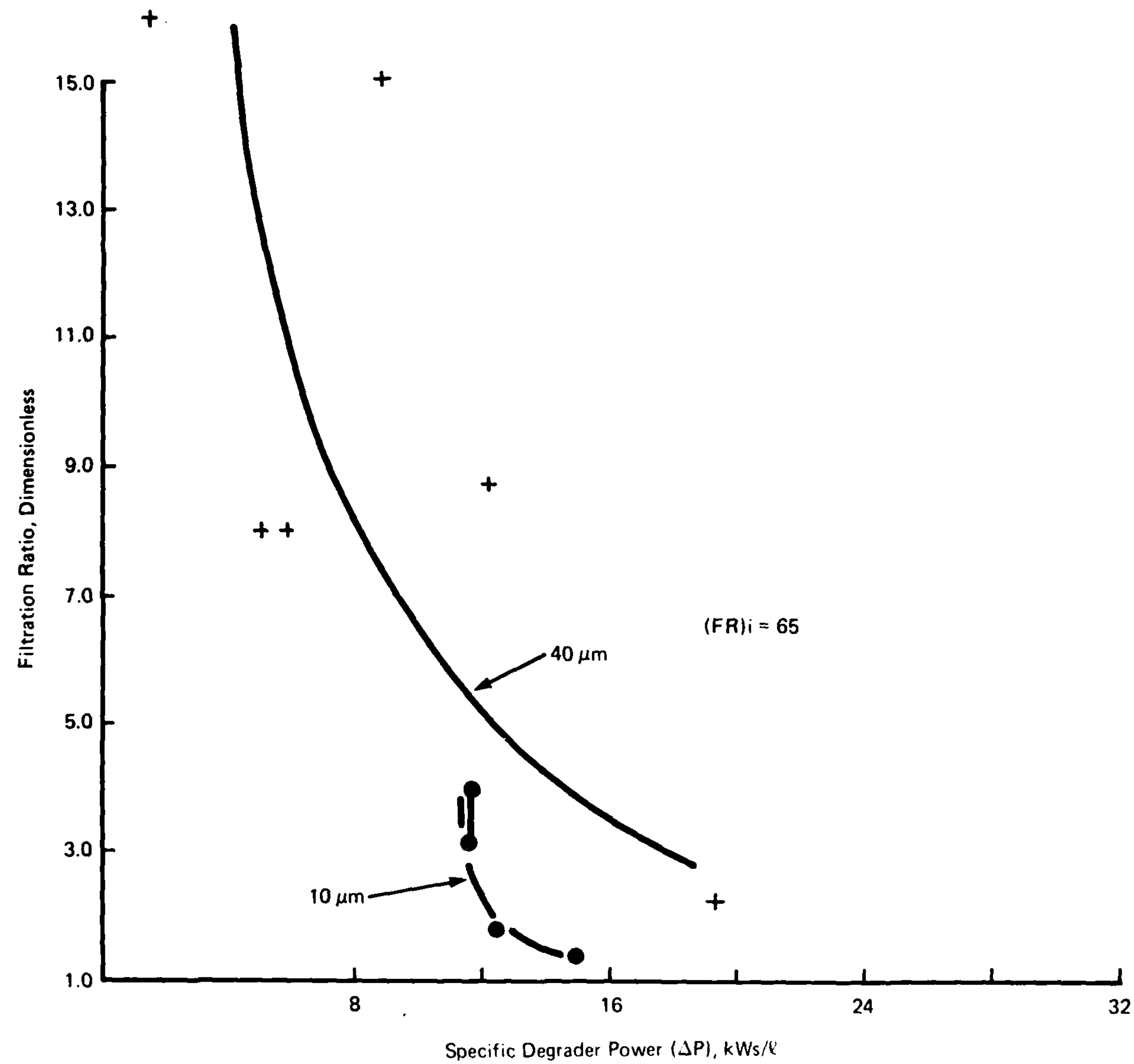

FIGURE 11. EFFECT OF SPECIFIC DEGRADER POWER THROUGH METAL SCREENS ON THE FILTRATION RATIO

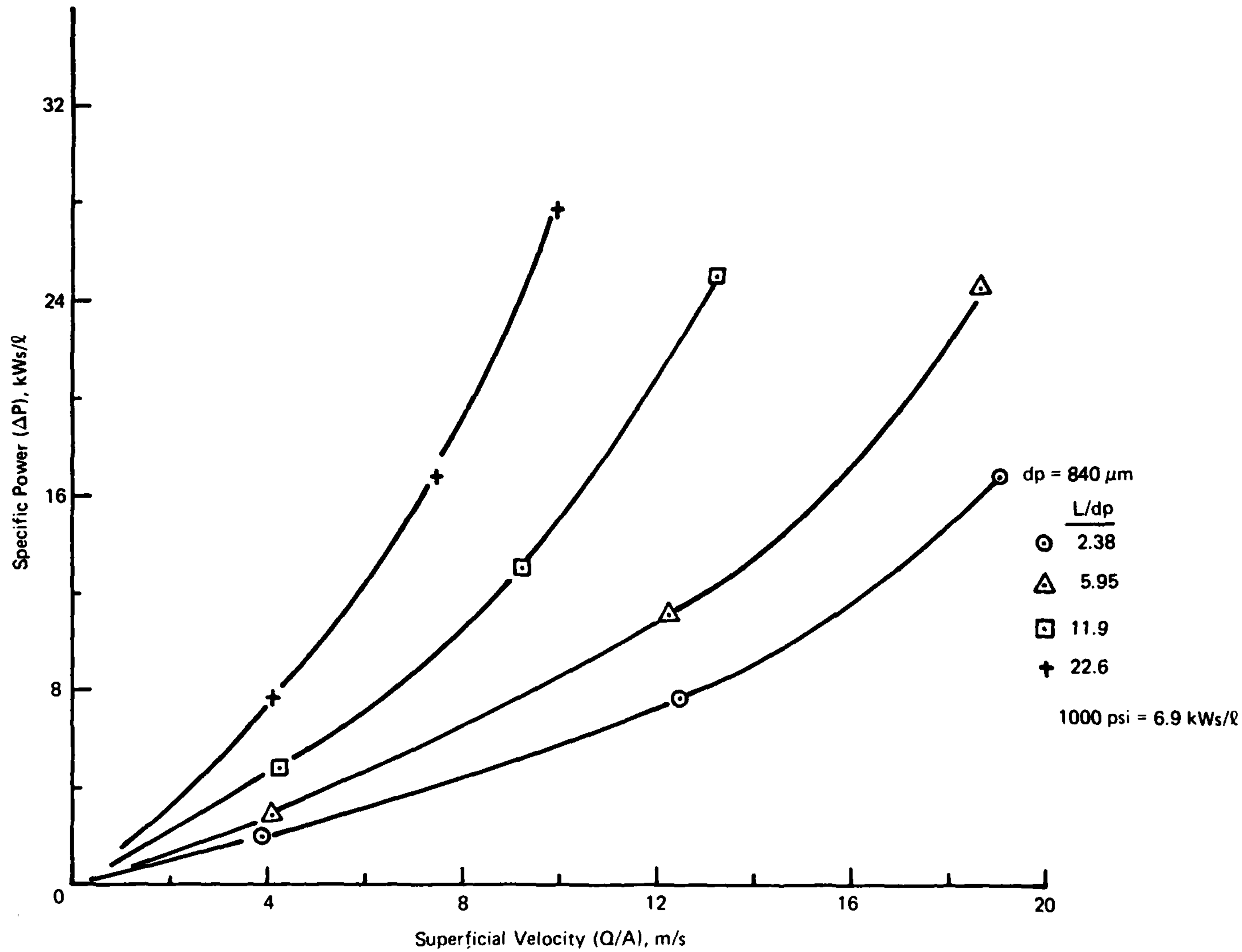

FIGURE 12. FLOW OF AMK THROUGH DIFFERENT LENGTH PACKED TUBES

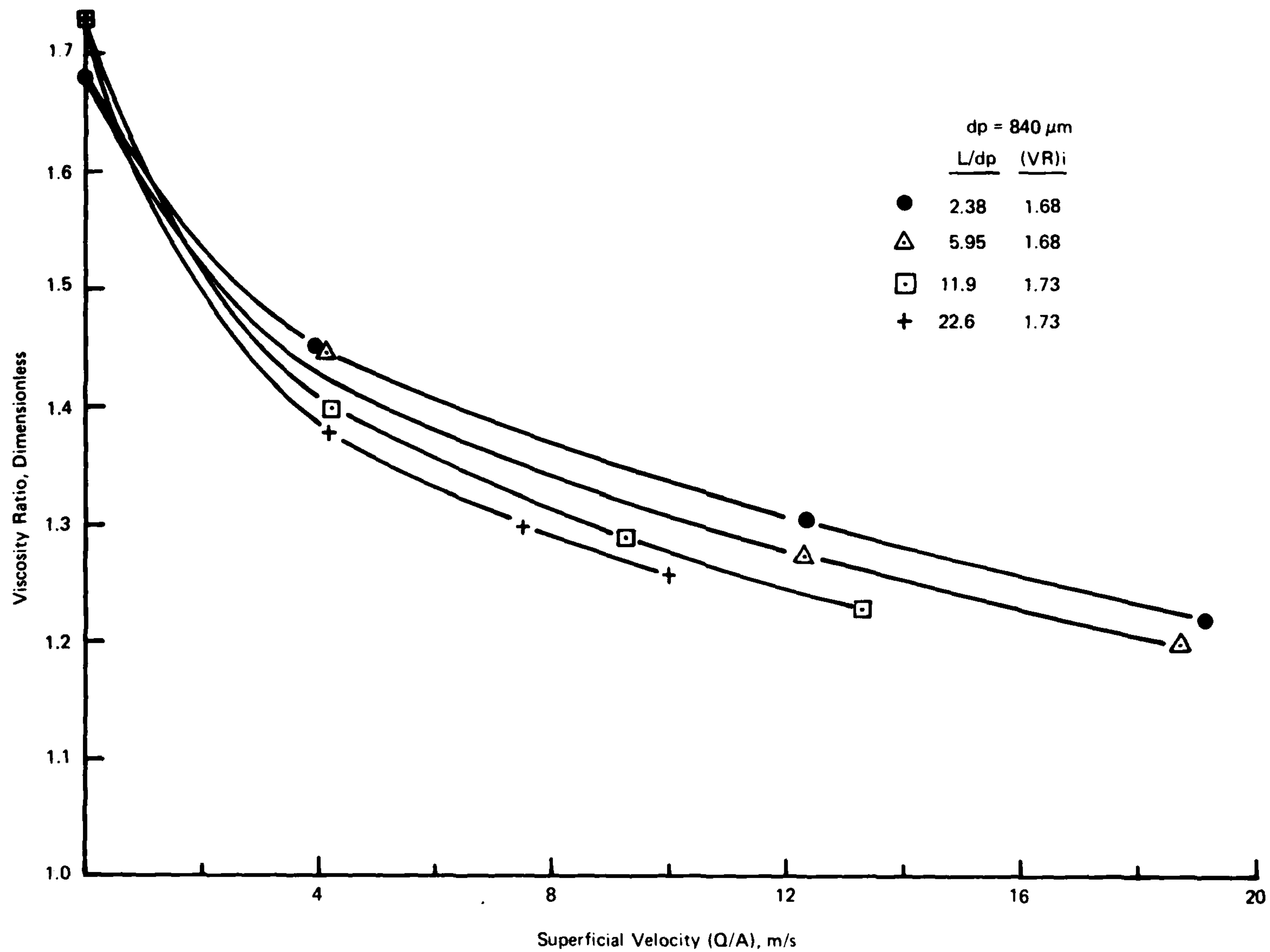

FIGURE 13. EFFECT OF TUBE LENGTH AND SUPERFICIAL VELOCITY ON THE VISCOSITY RATIO

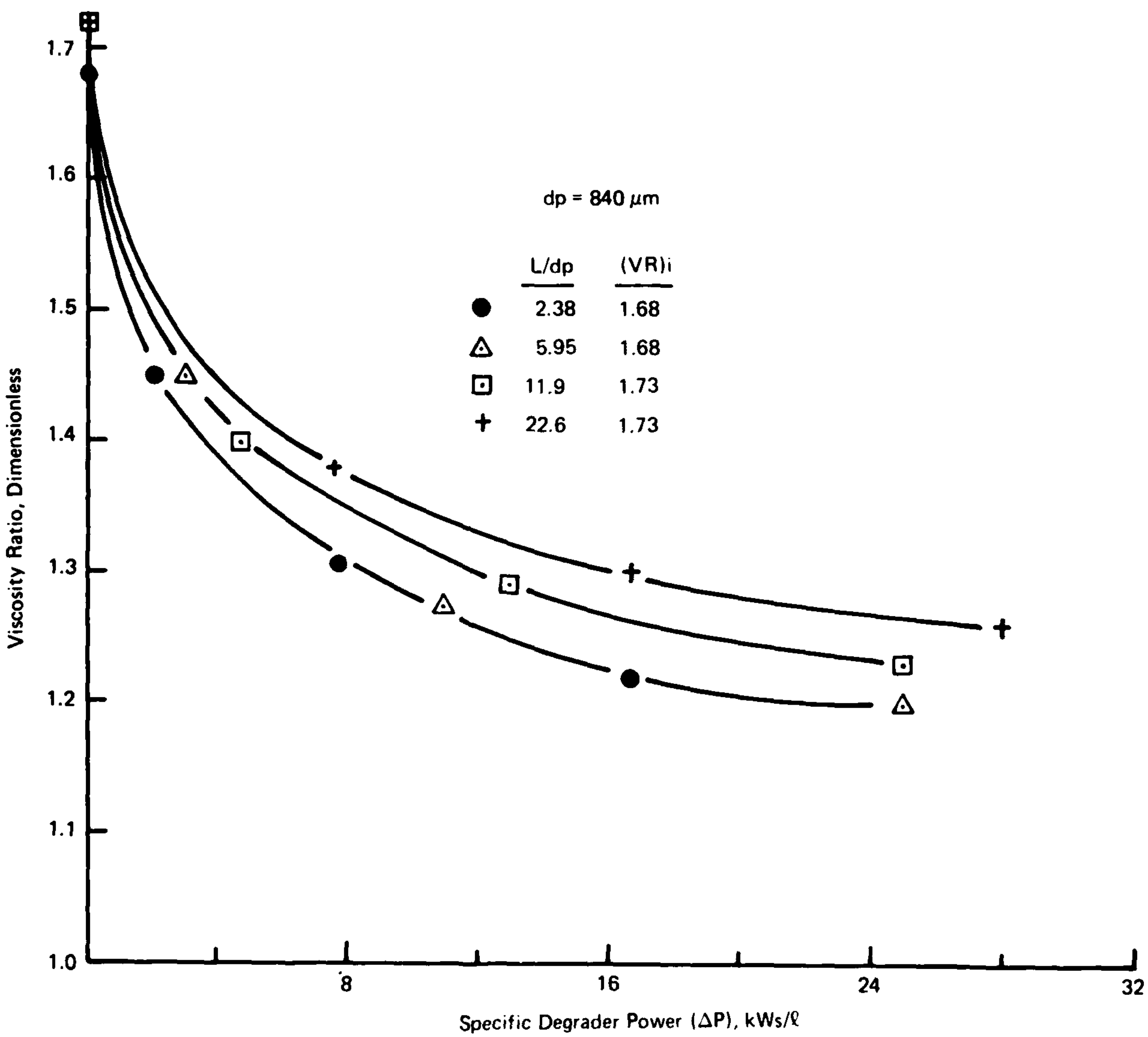

FIGURE 14. EFFECT OF TUBE LENGTH AND SPECIFIC DEGRADER POWER ON THE VISCOSITY RATIO

The results shown in Figure 15 indicate that increasing the tube length (L/dp) has the same general effect of reducing the superficial velocity required to produce a particular filtration ratio as was shown in Figure 13 for the viscosity ratio. While earlier results for AMK degraded with screens produced filtration ratios as low as 1.3, the lowest filtration ratio for the packed tube experiments shown in Figure 16 was 2.0. Although filtration ratios as low as 1.0 have been reported for AMK after it had been processed by the UK degrader, these low values are partly due to the fact that the fuel is at an elevated temperature (50° to 60°C above ambient). For example, results with the latest model UK degrader (D70) indicate that the filtration ratio of AMK processed at 75 kWs/l was approximately 1.0 at elevated temperatures and increased to approximately 2.0 after the fuel was cooled to ambient.[11] Increasing tube length generally lowers the superficial velocity required to produce the same filtration ratio; however, as with the viscosity ratio, a longer tube requires a higher specific degrader power (Figure 16).

Similar experiments were conducted with smaller glass beads (420 micrometer) and two different length tubes. Except for one important difference, the results of these experiments are in good agreement with those obtained with the larger beads (840 micrometer). This difference suggests that degradation with smaller beads is more efficient. For example, the results of tube length on the specific degrader power required to reduce the viscosity ratio to a particular value was almost identical for both sized beads. However, the effect of bead size on filtration ratio (Figure 17) was significant. In particular, the specific degrader power required to produce a filtration ratio of 2.0 was approximately twice as high for the larger beads (24 kWs/l compared to 12.5 kWs/l). Additional experiments to determine the effect of bead size were conducted with a sample of AMK blended by ICI Americas, Inc. The results of these experiments (Figure 18) substantiate that less power is required to reduce the filtration ratio to a specified level with the 420-micrometer beads than with 840-micrometer beads.

The mist flammability data for standard AMK (0.3% FM-9 + carrier fluid) degraded at different power levels and with different sized beads are presented in Figure 19. Below a velocity of 72 m/s, flame propagation of undegraded AMK is negligible compared to Jet A. However, at higher velocities flame propagation increases with disc velocity. It is important to note that this critical disc velocity is close to the pass/marginal air velocity for AMK made with carrier fluid in fuel spillage tests conducted at FAA Technical Center.[2]

At low degrader power (3 to 4 kWs/l), the critical disc velocity is only slightly lower than undegraded AMK; however, at 15 kWs/l and higher, the mist flammability of AMK is indistinguishable from Jet A. It should be noted that while the smaller beads were more effective in reducing the filtration ratio, no effect of bead size was evident for either the viscosity ratio or the mist flammability.

Ignition experiments utilizing a T-63 combustor rig indicate that degraded AMK requires only a slightly higher fuel-flow rate than Jet A to produce an ignition delay of 1 second or less:

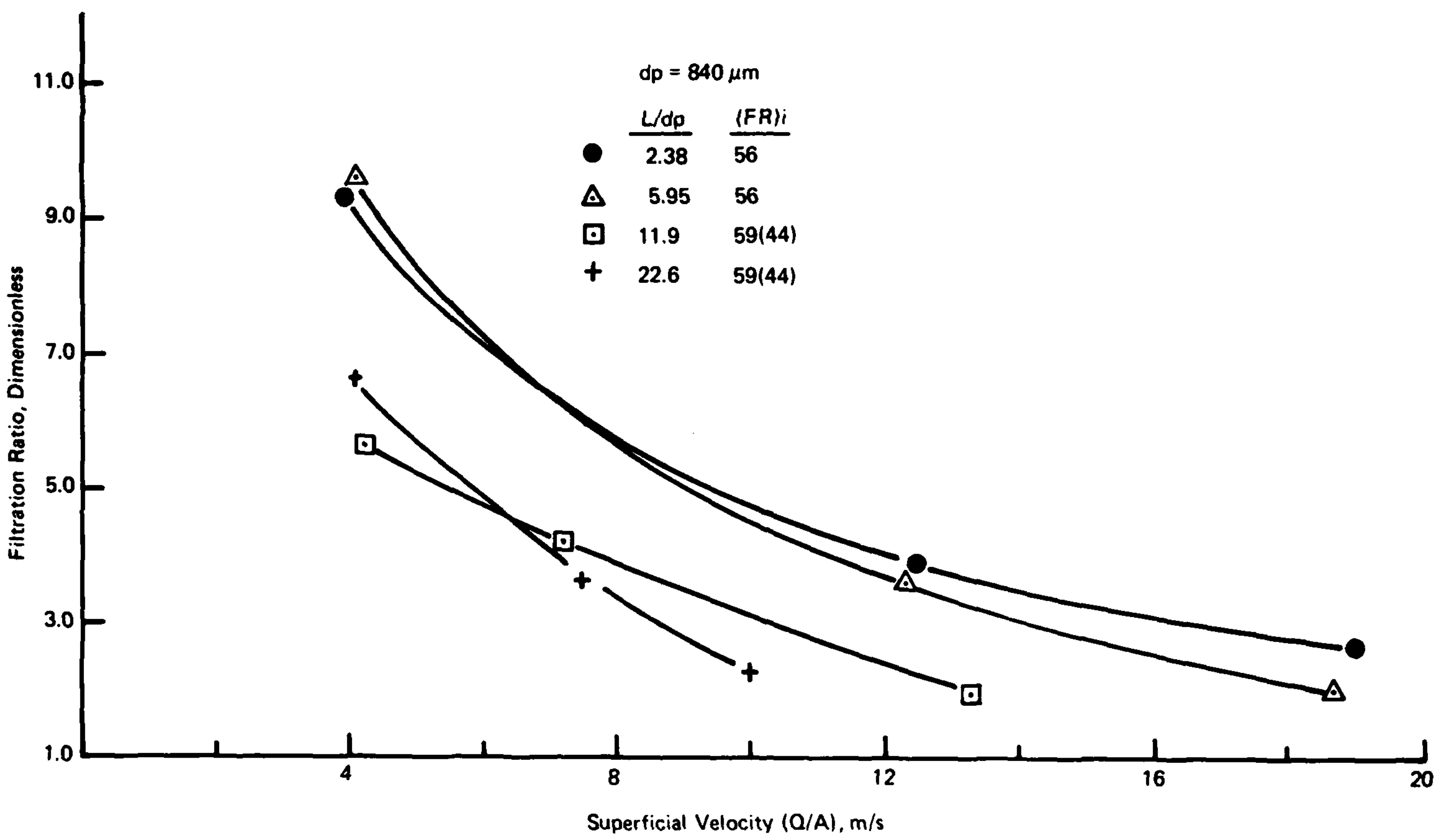

FIGURE 15. EFFECT OF TUBE LENGTH AND SUPERFICIAL VELOCITY ON THE FILTRATION RATIO

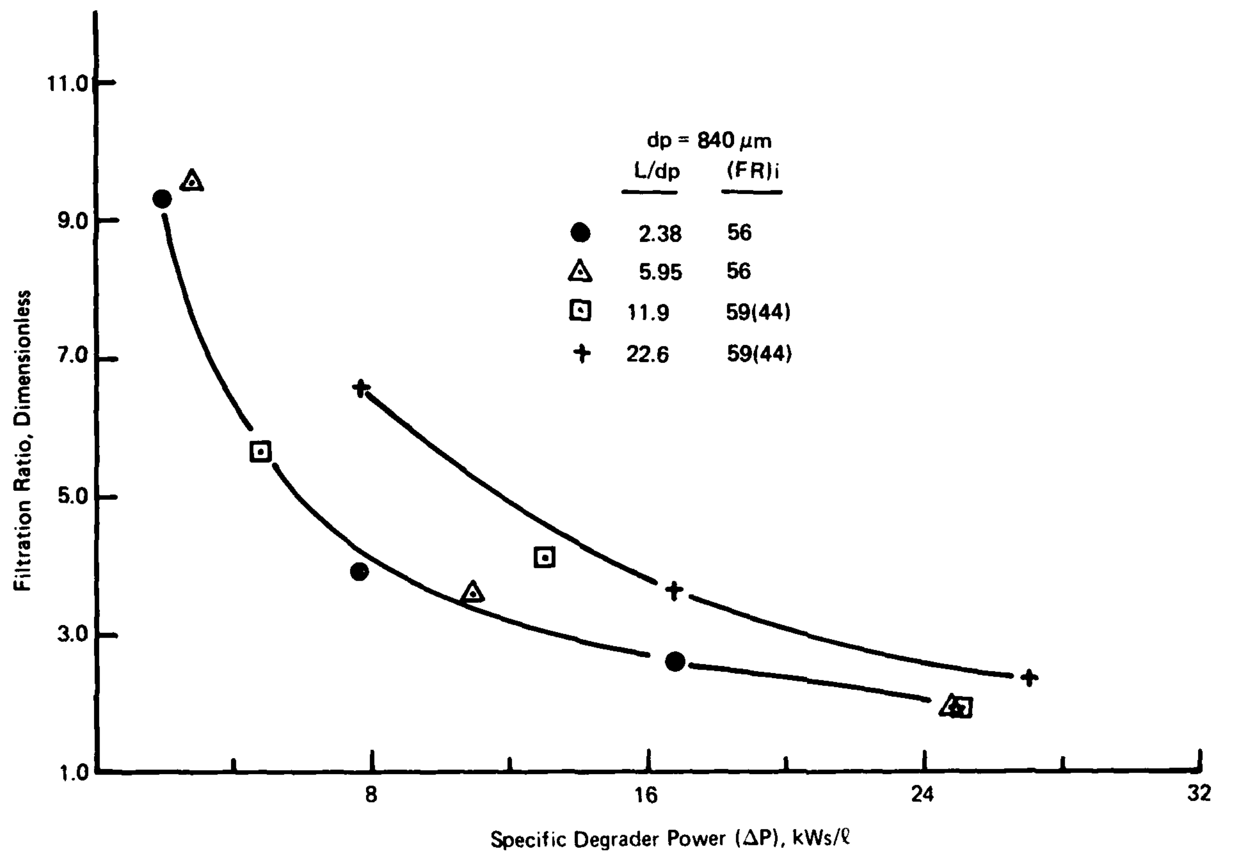

FIGURE 16. EFFECT OF TUBE LENGTH AND SPECIFIC DEGRADER POWER ON THE FILTRATION RATIO

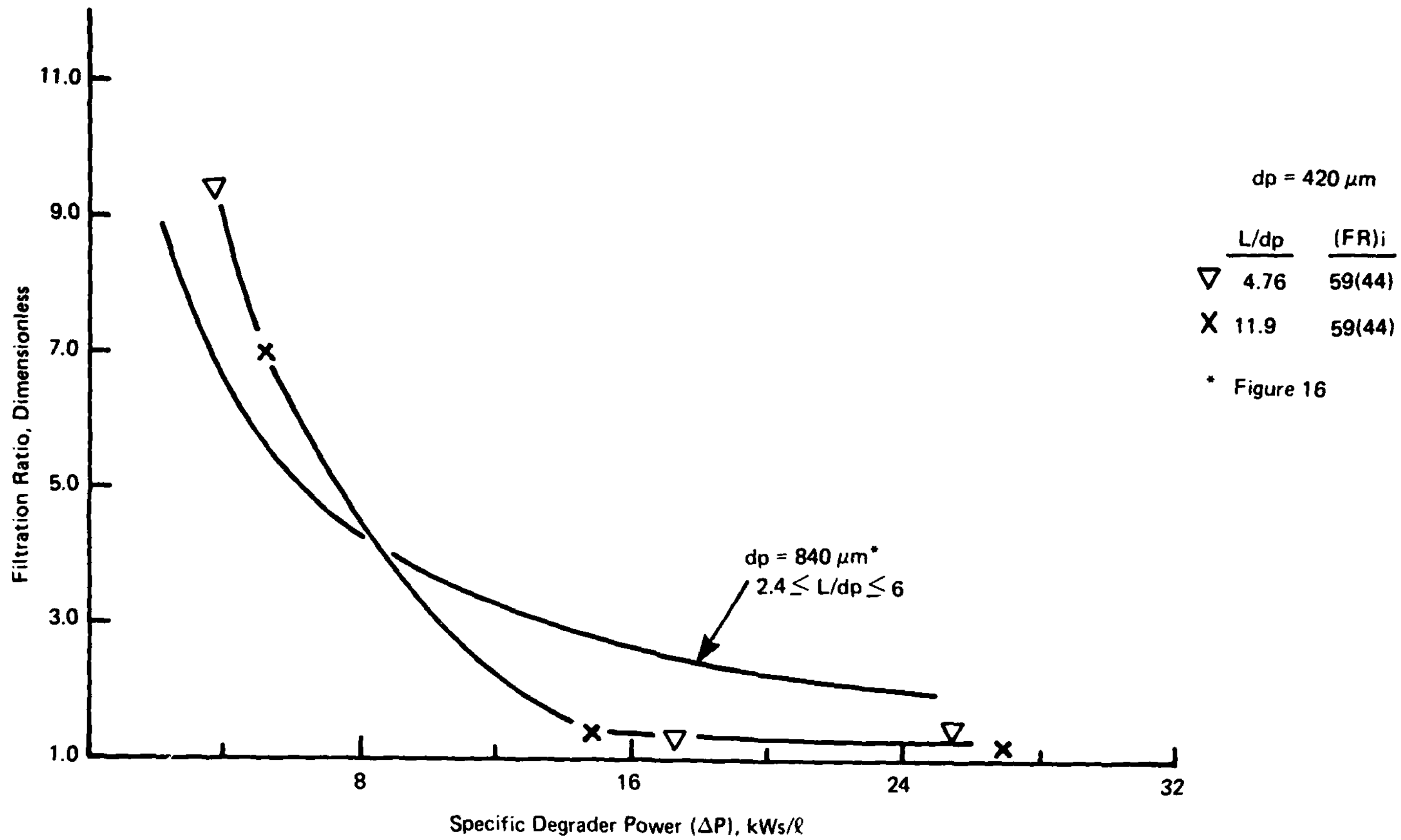

FIGURE 17. EFFECT OF BEAD SIZE AND SPECIFIC DEGRADER POWER ON THE FILTRATION RATIO

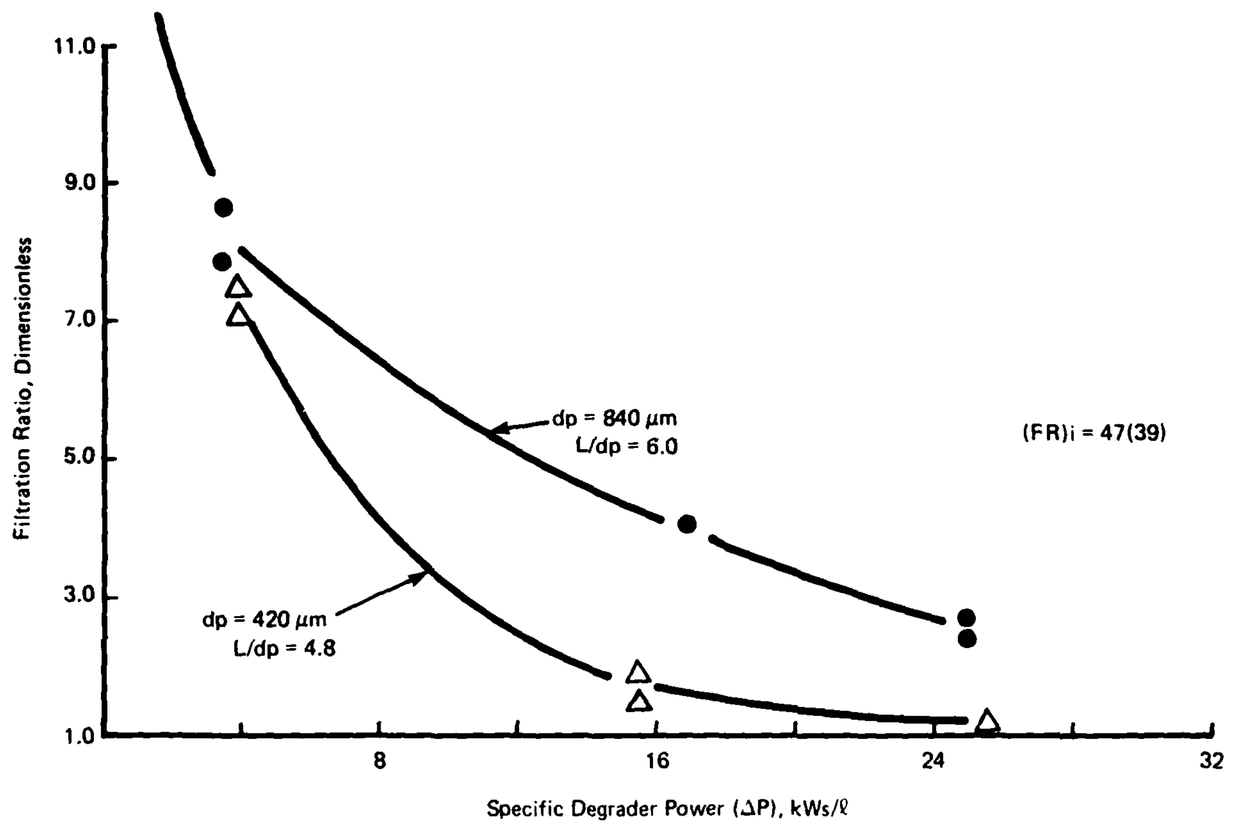

FIGURE 18. EFFECT OF SPECIFIC DEGRADER POWER ON THE FILTRATION RATIO OF AMK BLENDED BY ICI AMERICAS INC.

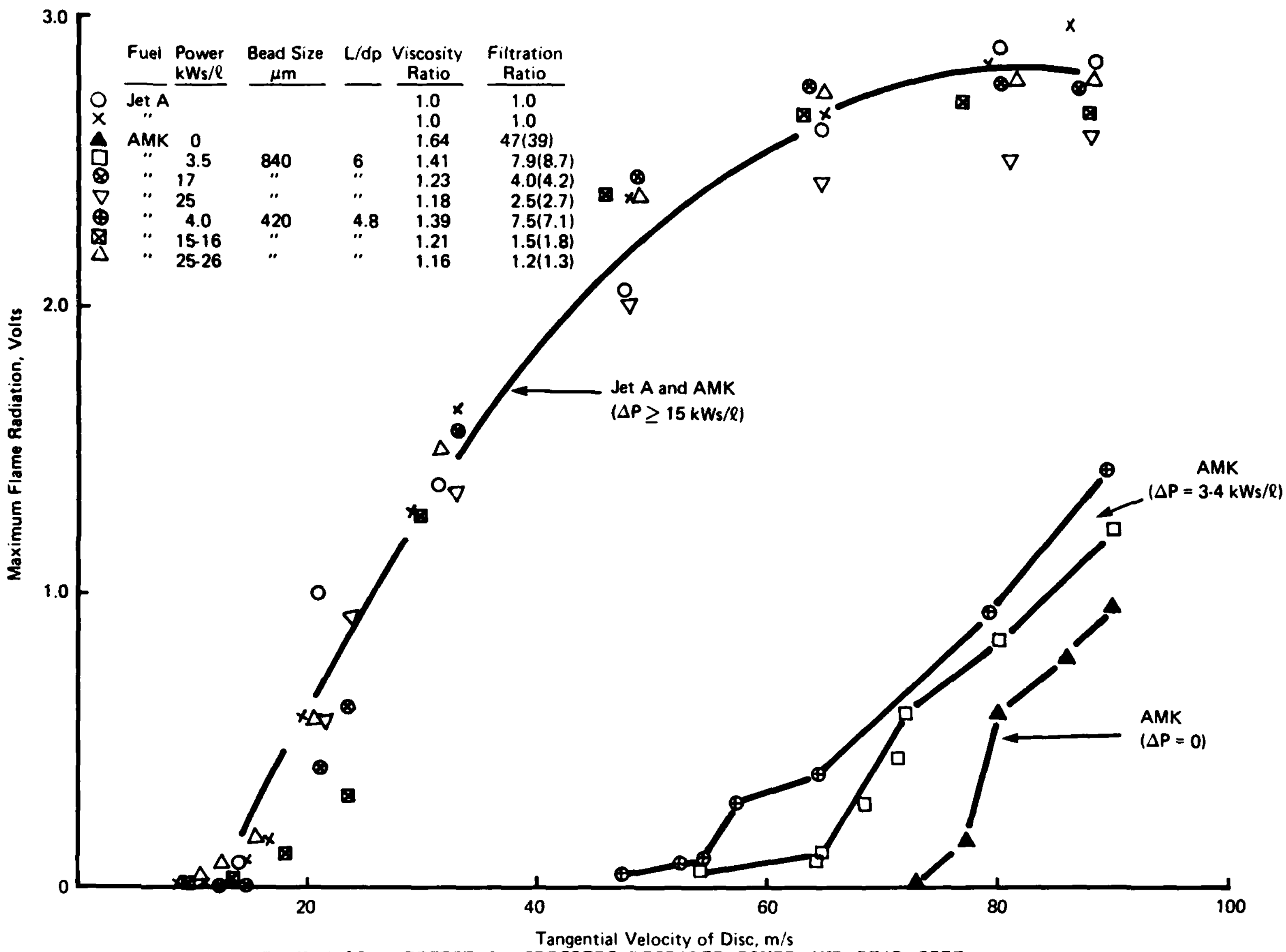

FIGURE 19. EFFECT OF SPECIFIC DEGRADER POWER AND BEAD SIZE ON MIST FLAMMABILITY OF AMK BLENDED BY ICI AMERICAS, INC.

Fuel	dp, μm	L/dp	ΔP, kWs/l	W*, lb/min
Jet A	--	--	--	0.41–0.45
AMK	420	12	28	0.55–0.58
AMK	420	12	28	0.50–0.54
AMK	420	6	26	0.51–0.53
AMK	840	6	26	0.56–0.59

* Burner inlet pressure - 20 psia, temperature - ambient;
air flow rate - 0.4 lb/s.

Although these are only preliminary results of relatively few experiments, they suggest that the spinning disc experiment is a reasonably good measure of the ignition resistance of degraded AMK.

Since the carrier fluid reduces the gel-forming tendency of AMK in Jet A, experiments were conducted to determine the effect of the carrier fluid on specific degrader power required to eliminate filtration and mist ignition resistance. While only small differences were observed in the power required to reduce the filtration resistance of AMK made with (Figures 17 and 18) and without (Figure 20) carrier fluid to a low value (i.e., FR<2), much larger differences were noted in the mist flammability (Figure 21). Despite the fact that the critical velocity of undegraded AMK without carrier fluid is 15 to 20 m/s lower than standard AMK, a much higher power level is required before any significant reduction in the critical velocity can be detected. For example, at a power level of 3 to 4 kWs/l, the critical velocity of AMK with carrier fluid was reduced from 73 to between 48 to 52 m/s (Figure 19). However, at power levels up to 8 kWs/l, the critical velocity of AMK without carrier fluid remained essentially constant. Furthermore, at a power level of 15 kWs/l or greater, the mist flammability of AMK with carrier fluid was indistinguishable from Jet A (Figure 19); however, the AMK without carrier fluid required 24 kWs/l to achieve this same condition.

Since AMK made with glycol but without amine produces no visible gel and yet appears to be as fire-resistant as standard AMK, experiments were conducted to determine the effect of the amine on specific degrader power. While the power level to produce filtration ratios in the range of 3 to 5 was much lower than for standard AMK (Figure 22), slightly higher power was required to obtain filter ratios below 2. Furthermore, while there was little or no effect of bead size on filtration ratio, changes in mist flammability were much more evident. For example, the results in Figure 23 show that the critical velocity of undegraded AMK (without amine) was approximately 76 m/s. At a power level of only 2.6 kWs/l, this was reduced to 58 m/s and at 5.2 kWs/l to approximately 40 m/s. At powers of 10 kWs/l and higher, the mist flammability of AMK without amine was essentially the same as Jet A. This higher susceptibility to mechanical degradation may be the reason why AMK made without amine behaves differently in different flammability tests.

E. Laminar Flow of AMK Through Porous Media

The laminar flow of dilute polymer solutions through porous media is often characterized by two distinctly different flow regimes. Below a critical strain rate, the flow resistance is determined by the shear viscosity; however,

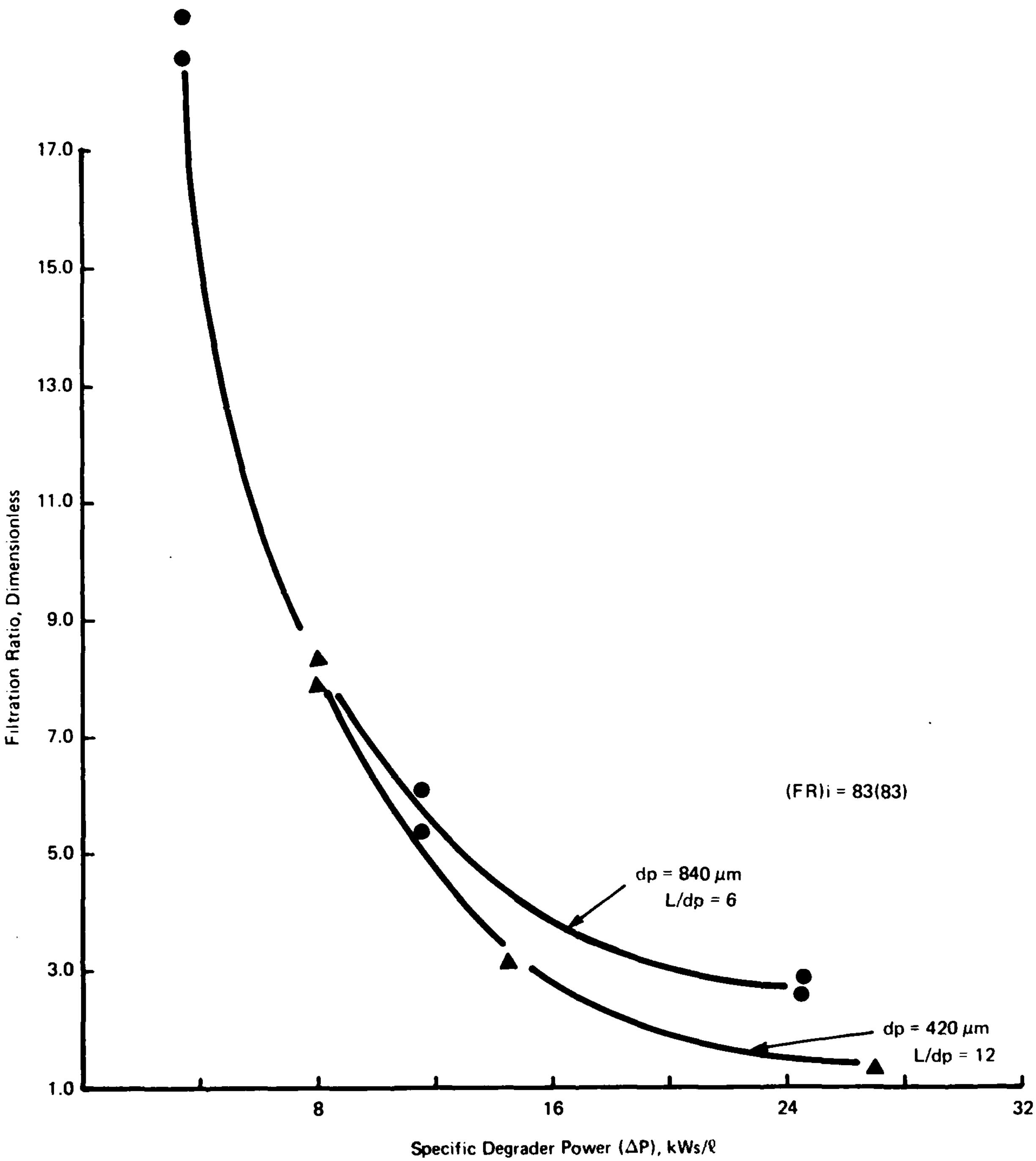

FIGURE 20. EFFECT OF SPECIFIC DEGRADER POWER ON THE FILTRATION RATIO OF AMK WITHOUT CARRIER FLUID

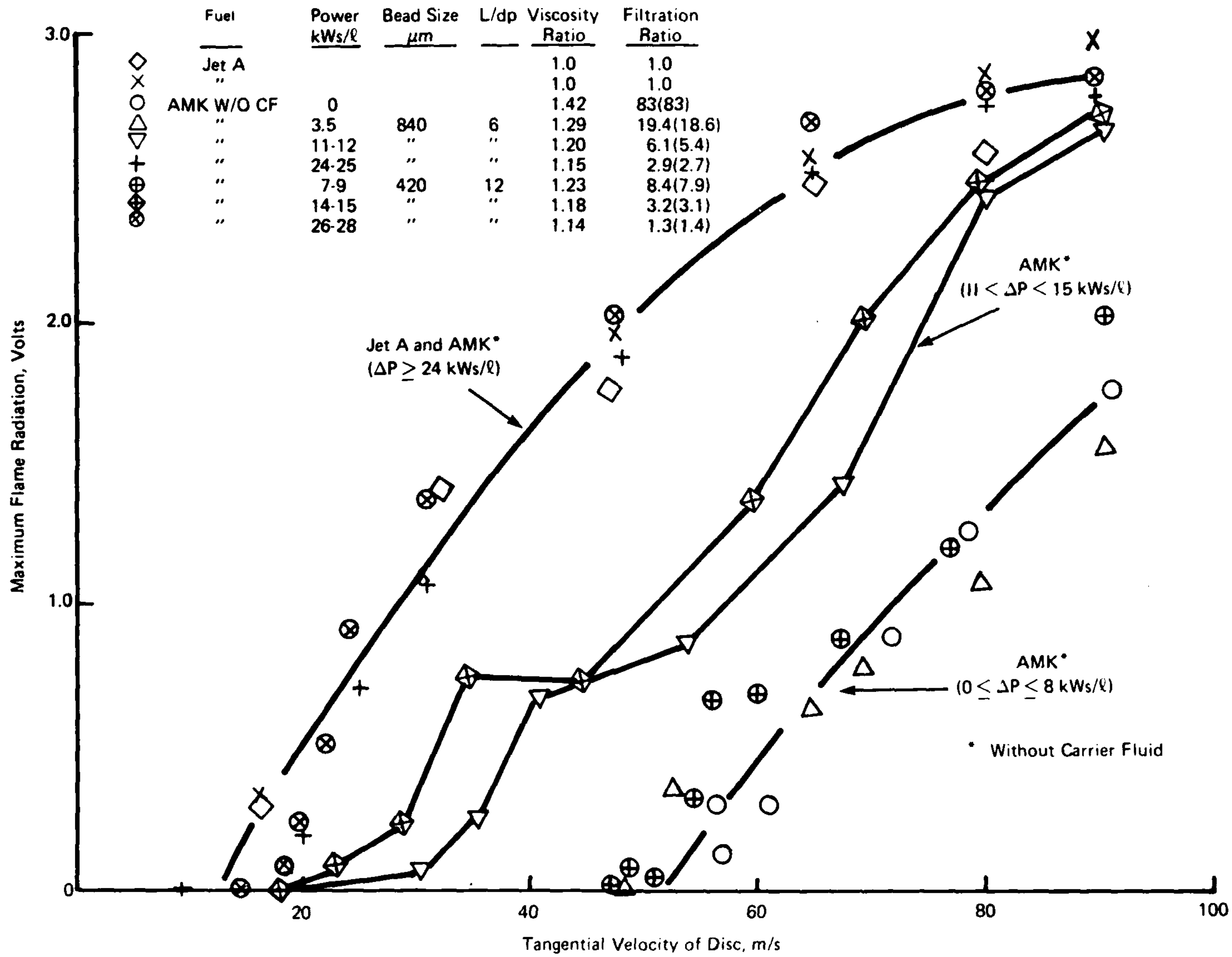

FIGURE 21. EFFECT OF SPECIFIC DEGRADER POWER AND BEAD SIZE ON MIST FLAMMABILITY OF AMK WITHOUT CARRIER FLUID

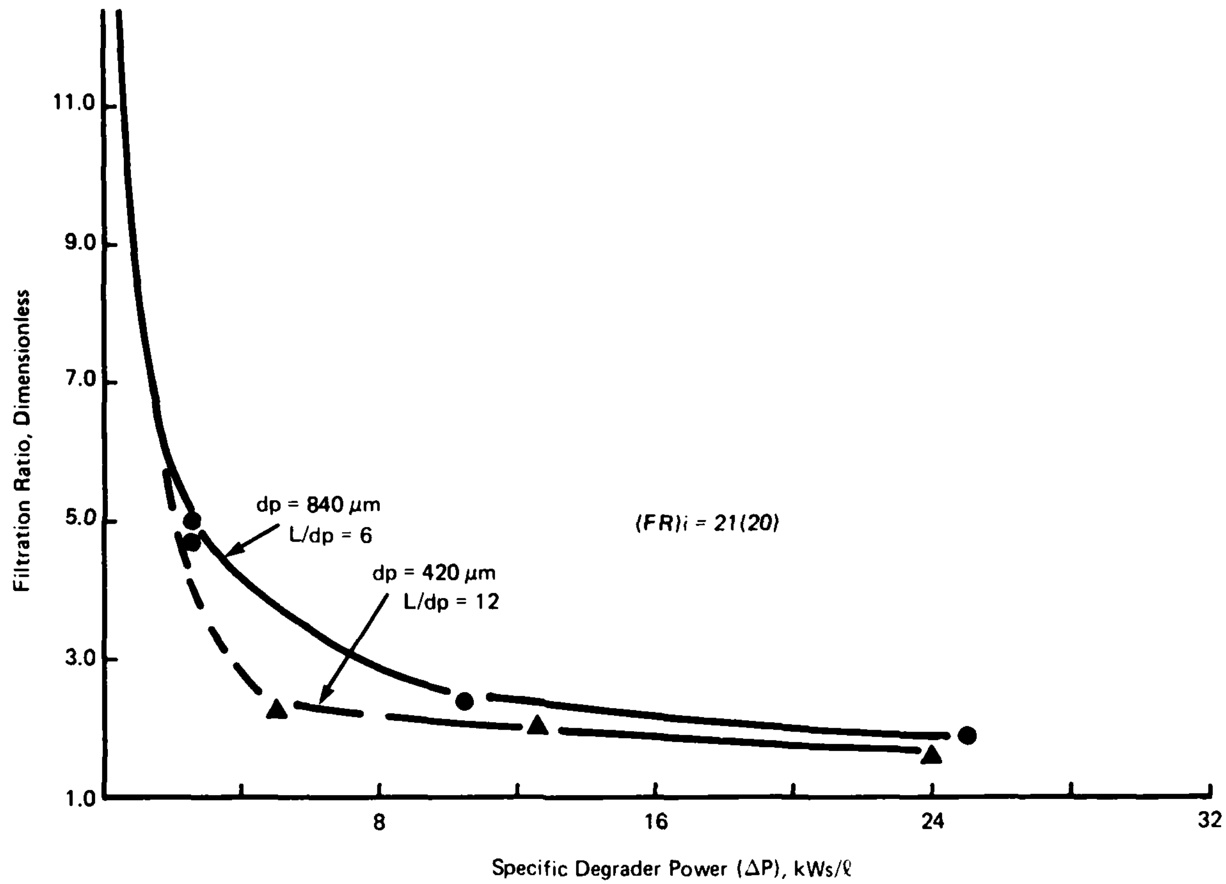

FIGURE 22. EFFECT OF SPECIFIC DEGRADER POWER ON THE FILTRATION RATIO OF AMK WITHOUT AMINE

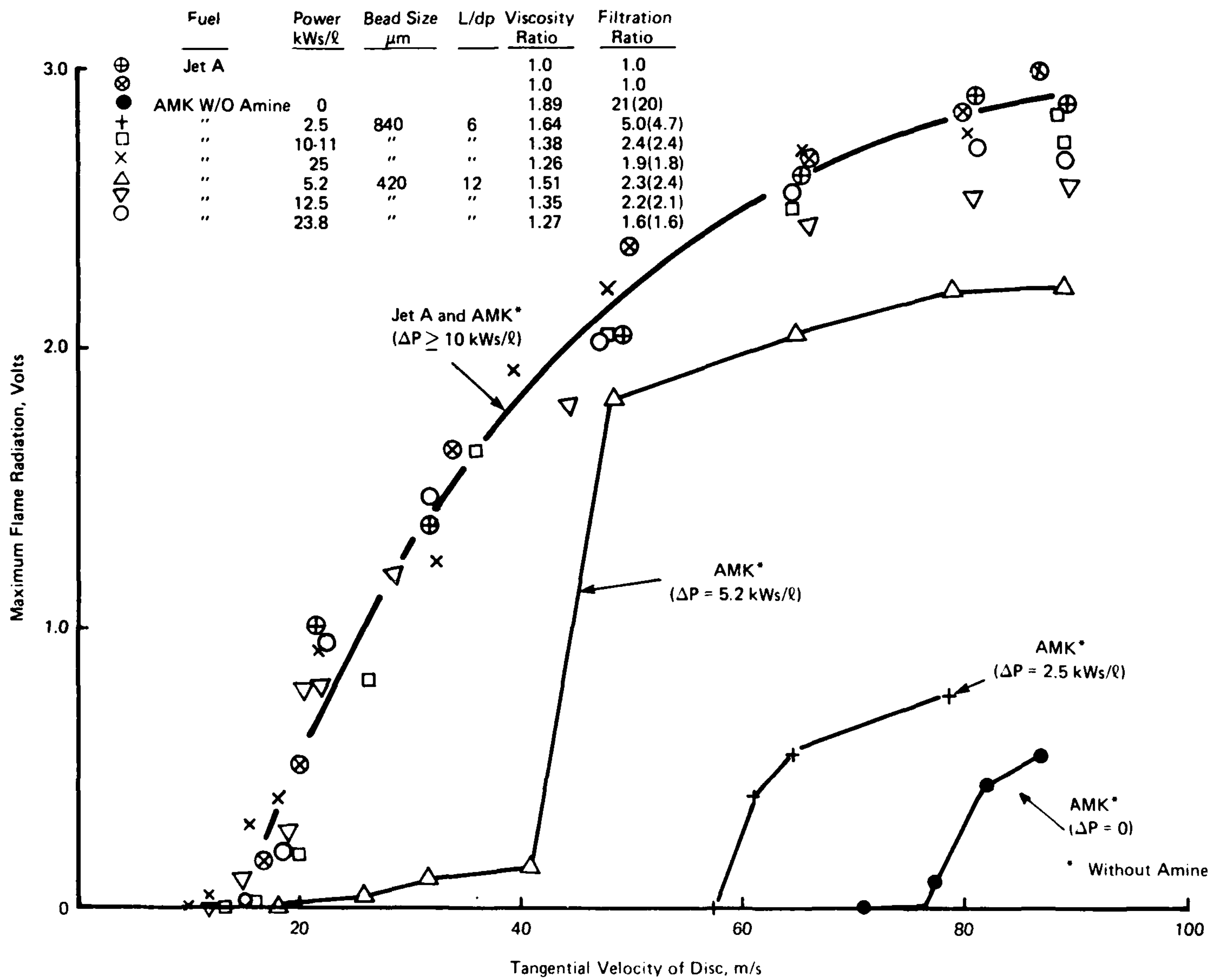

FIGURE 23. EFFECT OF SPECIFIC DEGRADER POWER AND BEAD SIZE ON MIST FLAMMABILITY OF AMK WITHOUT AMINE

at higher rate of strain the flow resistance increases dramatically and can become orders of magnitude higher than the shear viscosity.(5) This high-resistance flow regime is viscoelastic phenomenon associated with the elongational flow field that is characteristic of porous media.(5) Because of the gel-forming tendency of the FM-9 polymer, it is difficult to differentiate between this inherent viscoelastic phenomenon and filter plugging with the standard filtration test. Consequently, experiments were conducted to determine the effect of different pressure gradients on the velocity of Jet A and AMK through porous media, such as metal screens and filter paper.

The results in Figure 24 demonstrate the flow characteristics of Newtonian liquids, in this case Jet A and diesel fuel. Measurements were taken at increasing and decreasing pressures to detect filter plugging. It is interesting to note that the superficial velocity for Jet A at a pressure head of 20 cm is approximately 4 cm/s. This is close to the average superficial velocity* of 4.4 cm/s for Jet A in the standard filtration test. This illustrates the similarity in flow conditions between the standard filtration test and this experiment.

The linear flow characteristics shown in Figure 24 are representative of the laminar flow regime in which the Darcy Equation is valid:

$$V = \frac{k\Delta P}{\mu L} \qquad (1)$$

where $V = Q/A$ is the superficial velocity, ΔP is the pressure drop, L is the filter thickness, μ is the absolute (shear) viscosity, and k is the permeability.

More general relationships for non-Newtonian behavior have been derived (12), but for now this simple relation will be used. Since the permeability is a function of several factors such as pore size, pore shape, and porosity or void fraction, the following comments will refer to a specific filter configuration, i.e., 16- to 18-micrometer, stainless steel, Dutch weave. In this case, the Darcy Equation predicts that the velocity ratio of Jet A relative to diesel fuel (i.e., the filtration ratio) will be 3.8 cp/1.5 cp = 2.53. This is very close to the measured value shown in Figure 24. Thus while there is a large difference between the filtration ratio (40 to 50) and viscosity ratio (1.65 to 1.75) for undegraded AMK, the Darcy Equation predicts that these two measurements will have a common asymptote (i.e., FR → VR) for highly degraded AMK. Furthermore, this result demonstrates that the standard filtration test is basically a screen viscometer. Filtration measurements for two highly degraded samples of AMK substantiate this prediction for a 16- to 18-micrometer screen but not for paper filters:

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* While the average superficial velocity ($\overline{V}$) is not reported in the standard filtration test, it can be calculated from the fuel volume (96 ml) and screen area (4.5 cm^2) i.e., $\overline{V} = 21.3/t$, where t is the flow time in seconds.

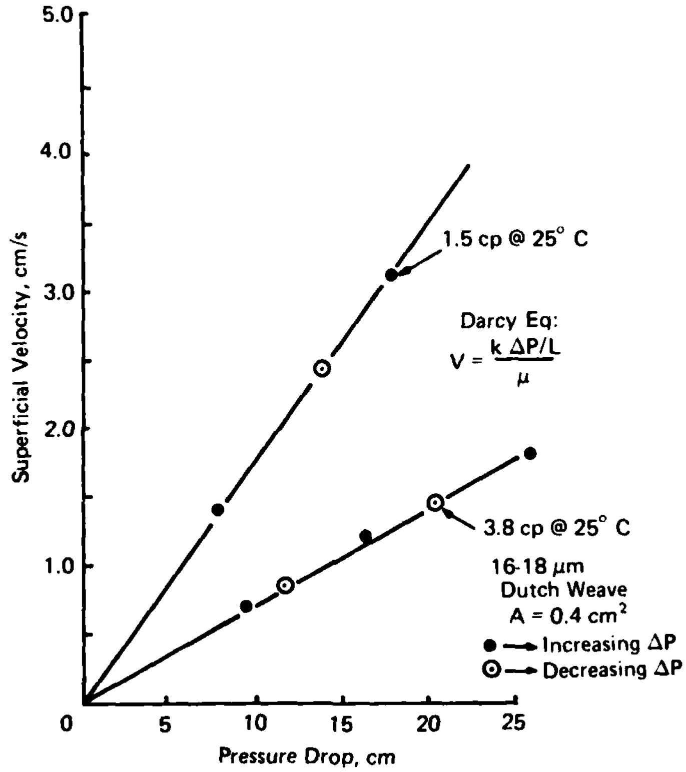

FIGURE 24. EFFECT OF PRESSURE DROP ON SUPERFICIAL VELOCITY OF NEWTONIAN LIQUIDS THROUGH METAL FILTERS

Viscosity Ratio	Filtration Ratio 16-18-μm Metal	20-μm Paper	10-μm Paper
1.17*	1.1(1.2)	8.6(13.6)	27(32)
1.22**	1.3(1.2)	3.4(3.2)	33(33)

* FM-9 with carrier fluid in Jet A, 25 kWs/l - SwRI degrader
** FM-9 with carrier fluid in Jet A-1, 100 kWs/l - RAE degrader

If these samples were fully degraded, the filtration ratio should be the same value as the viscosity ratio regardless of the filter material. Consequently, the increasing filter ratio with decreasing filter size indicates that the standard filtration test, which specifies a 16- to 18-micrometer metal screen, overestimates the level of polymer degradation. The reason for this will be explained by experiments with partially degraded AMK.

The flow characteristics of AMK degraded at different power levels are compared to those of Jet A (dashed line) in Figure 25. These results show that at these pressure gradients, undegraded AMK (VR = 1.69, FR = 40) cannot be made to flow at a velocity greater than 0.1 cm/s. While the hysteresis loop indicates a loss in permeability due to filter plugging, the magnitude of this effect depends on the value of the pressure gradient that is applied (i.e., there is little or no plugging at a pressure drop of 10 to 20 cm). This result suggests that even undegraded AMK can be filtered like a Newtonian fluid provided that the superficial velocity is below a critical value. For these experimental conditions, the critical velocity is approximately 0.1 cm/s; however, it is expected that the critical velocity will increase or decrease in direct proportion to the pore size of the filter. It is interesting to note that the maximum velocity in this test is almost identical to the average velocity for the sample of undegraded AMK in the standard filtration test (0.11 cm/s).

The critical velocity also depends on the level of specific degrader power. For example, at 5 kWs/l (VR = 1.29, FR = 7.9) the critical velocity is approximately 0.5 cm/s. Furthermore, the critical velocity is again very close to the average velocity for this degraded sample in the standard filtration test (0.56 cm/s). Increasing the specific power level to 9 kWs/l (VR = 1.2, FR = 2.9) increased the critical velocity to 1.0 cm/s; however, the average velocity in the standard test was 1.5 cm/s. The reason for the excellent agreement between these two tests for undegraded and slightly degraded AMK but poorer agreement as the degradation level increased was felt to be related to the induction time for gel formation. For example, rheological measurements indicate that the induction time is relatively rapid for undegraded AMK (<1 second), but significantly higher for degraded AMK.[13]

In order to test this hypothesis, filtration experiments were conducted with Jet A and a highly degraded AMK sample (VR = 1.2, FR = 1.2) at different pressure gradients and for different flow times. This particular AMK sample was degraded by RAE at a power level of 100 kWs/l. The results of these experiments (Figure 26) show no effect of flow time on the velocity of Jet A over the range of 0.5 to 3.0 minutes. However, different results were observed for the degraded AMK depending on the flow time. For relatively short times of 30 seconds or less, the superficial velocity of the degraded AMK increased

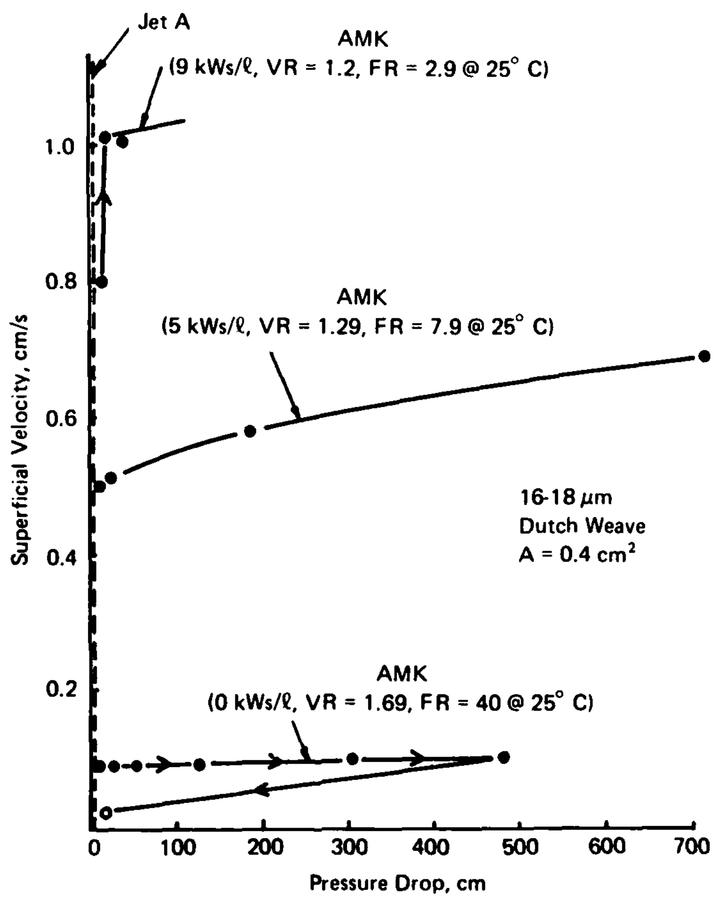

FIGURE 25. EFFECT OF PRESSURE DROP AND SPECIFIC DEGRADER POWER ON THE SUPERFICIAL VELOCITY OF AMK THROUGH METAL FILTERS

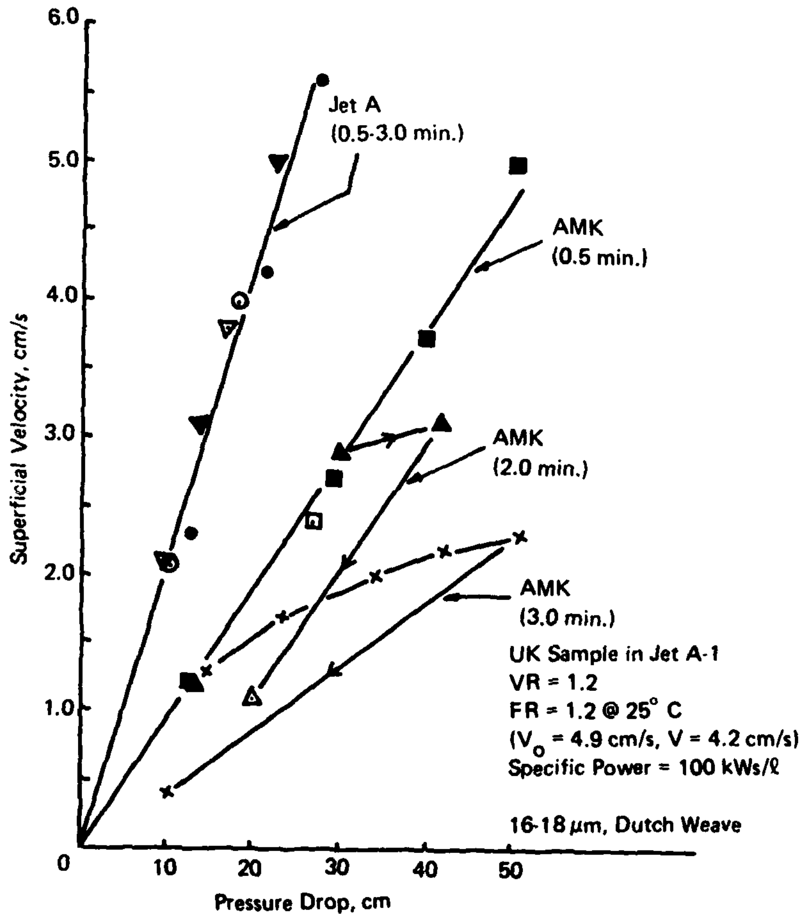

FIGURE 26. EFFECT OF PRESSURE DROP AND FLOW TIME ON THE SUPERFICIAL VELOCITY OF HIGHLY DEGRADED AMK THROUGH METAL FILTERS

linearly with increasing pressure drops. For a flow time of 2 minutes, a critical velocity is evident at 3 cm/s, and at a flow time of 3 minutes, the critical velocity was close to 1 cm/s. The hysteresis loops for flow times above 2 minutes clearly indicate that the critical velocity is associated with gel formation and filter plugging.

The results of these experiments confirm that the flow time (4 to 5 seconds) in the standard filtration test is too short to detect gel formation with highly degraded AMK. While the previously described experiment is capable of detecting these effects, the time-dependent nature of this phenomenon suggested the need for a filtration test that more closely simulated flow conditions in an aircraft fuel delivery system, and in particular one that is able to measure flow resistance as a function of time.

In the pump filtration test, a gear pump and drive transmission are used to force fuel through a filter at different flow rates. The pressure drop across the filter is measured as a function of time by a transducer and stripchart recorder. For the following experiments, the flow time was held constant at 2 minutes. Experiments conducted with Jet A (Figure 27) showed no measurable increase in pressure over the 2-minute interval. Furthermore, the pressure drop increased linearly with increasing superficial velocity (which is characteristic of laminar flow of a Newtonian liquid), and there was no evidence of hysteresis when the velocity was decreased. It is also important to note that at a velocity of 4 cm/s, the measured pressure drop was 20 cm. This is close to the average pressure drop that results in a velocity of 4 to 5 cm/s for Jet A in the standard filtration test.

Significantly different experimental results (Figure 27) were obtained with what should have been highly degraded AMK, as evidenced by the filtration ratio of 1.2 at 25°C. In this case, the pressure drop remained independent of time as long as the velocity was below 1 cm/s. It is important to note that the value of the filtration ratio is equivalent to an average velocity in the standard filtration test of close to 4 cm/s. At higher velocities, the pressure drop increased linearly with time after a short induction time. The effect of increasing pressure with time is illustrated by the separation of the data points at a fixed velocity. The rate of pressure rise with time was significantly higher as the superficial velocity increased above 1 cm/s. While this time effect appeared to be reversible in that the pressure drop was independent of time when the velocity was reduced below 1 cm/s, the hysteresis loop is definite evidence of filter plugging (i.e., ΔP = 5 cm at V = 0.9 cm/s for V increasing and ΔP = 100 cm at V = 0.9 cm/s for V decreasing). The results shown in Figure 28 indicate that even a sample of AMK degraded at very high power levels (100 kWs/l;RAE) exhibits time-dependent filter plugging at much lower velocities than would be expected from the standard filtration test.

The pump filtration test has been used to estimate the conditions for which AMK will filter like a Newtonian liquid. The critical velocity is determined by the highest velocity at which the pressure drop across the filter remains constant for 2 minutes. The results of preliminary experiments to determine the effect of surfactants, specific degrader power, degrader geometry, and filter material are summarized in Figures 29 and 30. For undegraded AMK, the critical velocity with the standard filter material (16- to 18-micrometer, stainless steel, Dutch weave) was between 0.07 and 0.09 cm/s. Because of the difficulty of working at velocities much lower

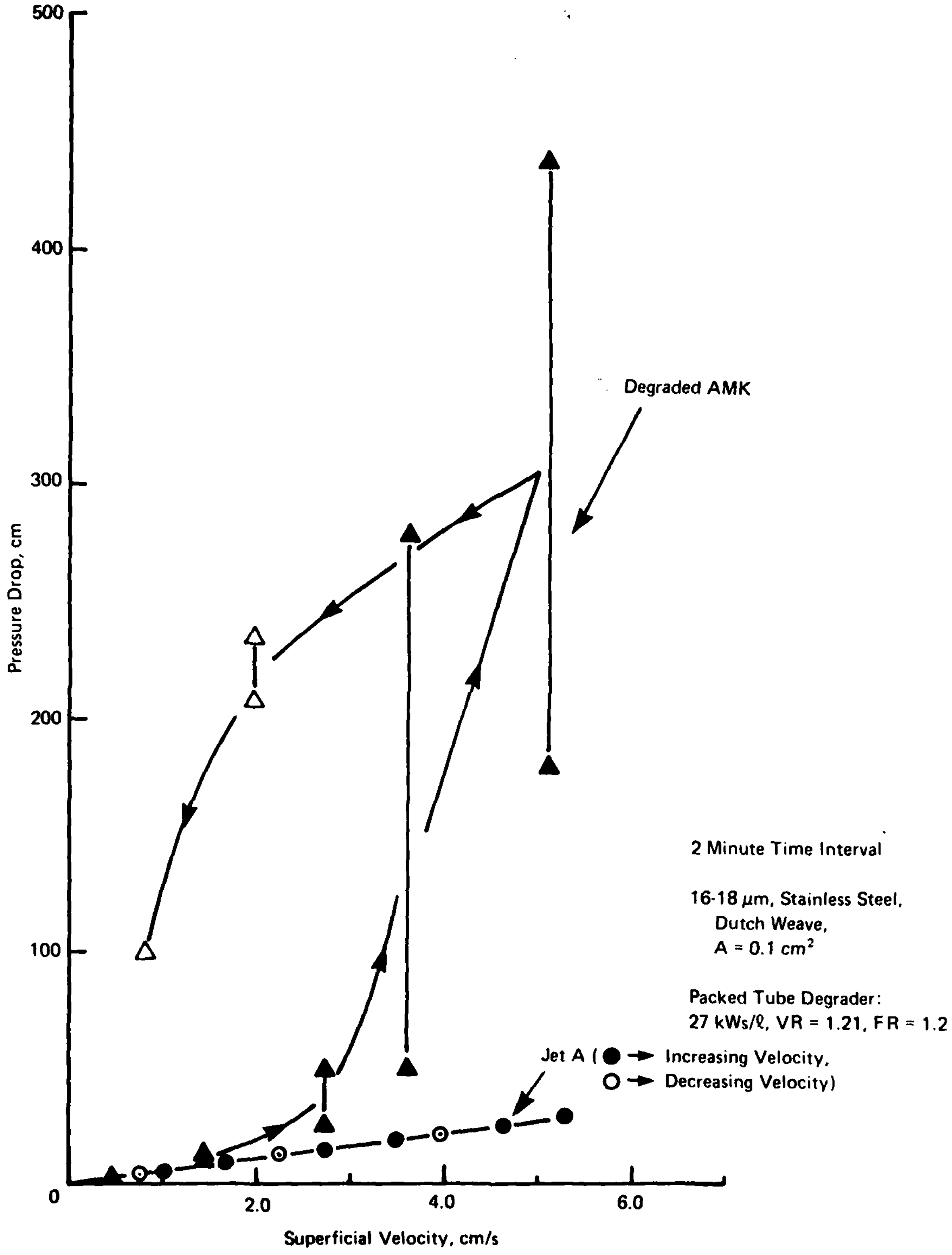

FIGURE 27. EFFECT OF SUPERFICIAL VELOCITY ON PRESSURE DROP ACROSS FILTER WITH JET A AND DEGRADED AMK

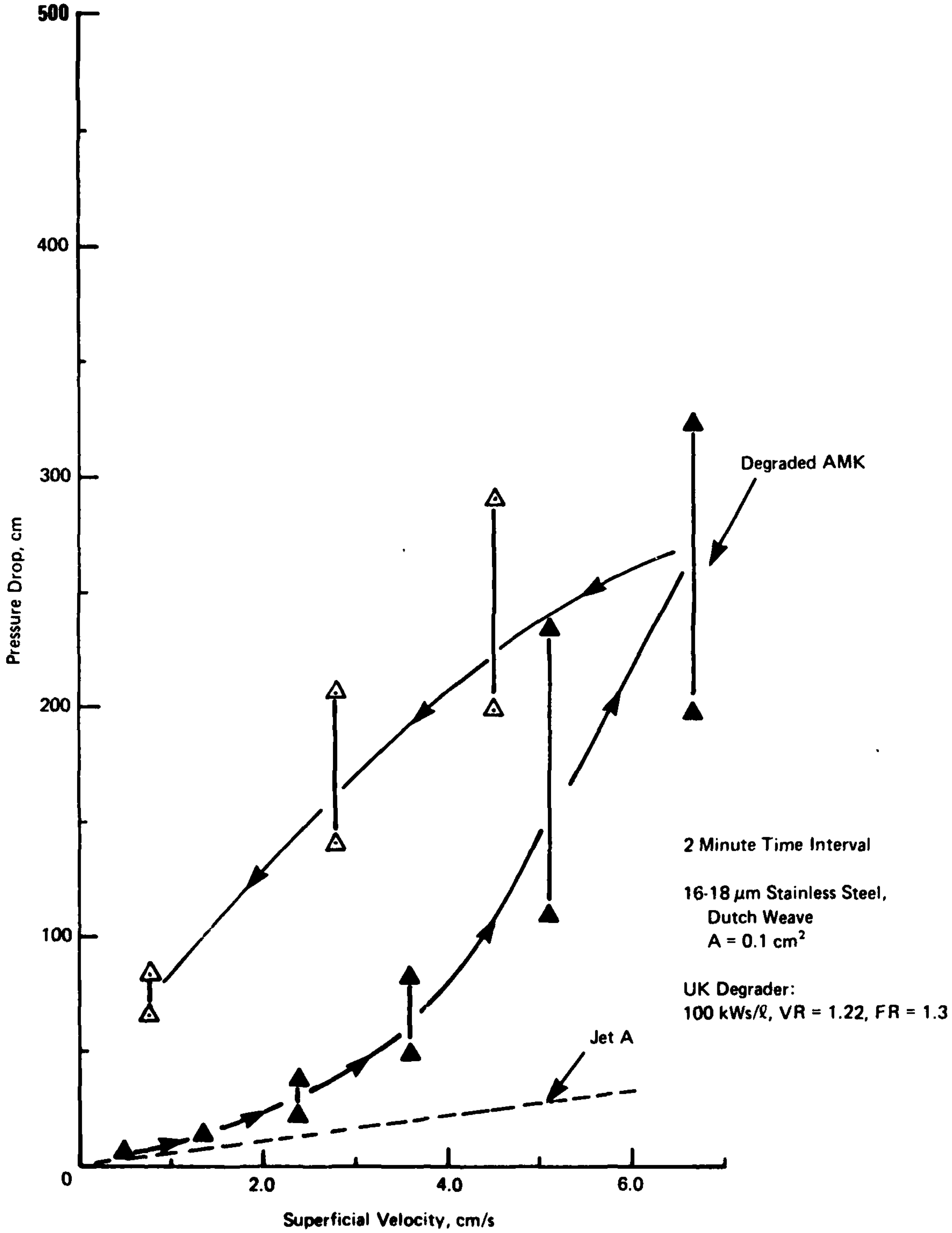

FIGURE 28. EFFECT OF SUPERFICIAL VELOCITY ON PRESSURE DROP ACROSS FILTER WITH JET A AND DEGRADED AMK

than 0.07 cm/s with the current experimental configuration, no attempt was made to measure the critical velocity for undegraded AMK with a paper filter (20 micrometers); however, it is expected to be approximately 0.03 to 0.05 cm/s.

The addition of surfactant (SO-A) increased the critical velocity of undegraded AMK in direct proportion to the amount added (Figure 29). It is particularly important to note that at 0.5% SO-A, the critical velocity increased to between 0.34 and 0.39 cm/s with the metal filter. Since this is essentially the same as the filtration velocity at takeoff for many commercial aircraft (15,000 lb/hr, 1000 sq in.), these results suggest that AMK/SO-A may filter like Jet A without mechanical degradation, if metal screens can be utilized. The slightly lower critical velocity for AMK/SO-A with the 20-micrometer paper filter is attributed to the reduced porosity and wider range of pore sizes that is typical for paper compared to metal filter material.

The effect of specific degrader power, degrader geometry (bead size), and filter material are presented in Figure 30. As expected, the results of these experiments indicate that the critical velocity increased with specific degrader power. However, bead size had a very unexpected effect. The results specific in Figure 30 show that for the metal filter, the larger beads (840 micrometers) are more effective in increasing the critical velocity. This is a reversal of the results discussed earlier in which it was found that the smaller beads (420 micrometers) required less power to reduce the filtra-tion ratio than the larger (840 micrometers) beads (*Figures* 17 *and* 18). These data also indicate that the highest critical velocity for AMK degraded through the larger beads occurs at an intermediate power level; however, this may be partly due to experimental accuracy. Very little effect of bead size was evident for the critical velocity of degraded AMK with the paper filter. Since those results differ from the observed effect of bead size on the filtration ratio, it is evident that previous experimental results involving geometric factors, such as L and L/dp on degradation, are subject to question. Consequently, it is recommended that degradation experiments should be repeated to determine the effect of degrader geometry on the critical filtration velocity.

F. Quality Control Tests For Polymer and Carrier Fluid Content

Numerous AMK samples that were blended by ICI Americas, Inc. and which had been used in fuel spillage tests at FAA Technical Center were analyzed for polymer content by a steam jet gum technique (ASTM D-381). The results of these tests are summarized in Table 1. Except for the first six samples, there is excellent agreement with the nominal polymer content assigned to the blend by ICI and the gum content.

Because of the suspected importance of the carrier fluid to mist flammability, efforts were initiated to analyze AMK for the amount of glycol and amine. The glycol analysis is based on infrared absorbance while the amine content is determined by combustion and chemiluminescence. Preliminary results appear promising in that a sample of AMK received from ICI Americas, Inc. was found to contain glycol and amine at concentrations very close to the expected values. However, additional experiments are required to establish repeatability accuracy, and the possible effects of other variables such as base fuel composition and hydrogen bonding with the FM-9 polymer.

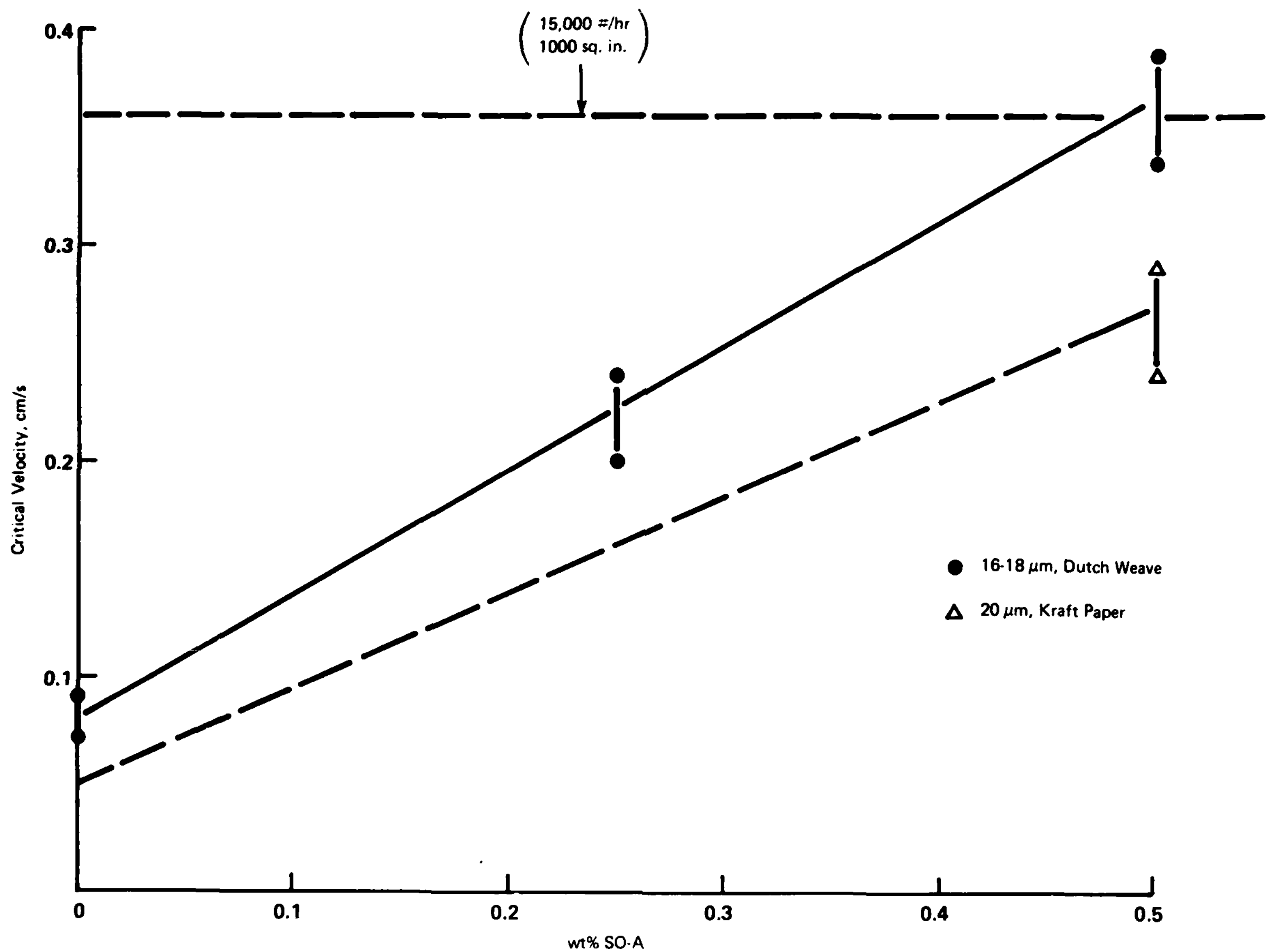

FIGURE 29. EFFECT OF SURFACTANT ON THE CRITICAL FILTRATION VELOCITY OF UNDEGRADED AMK

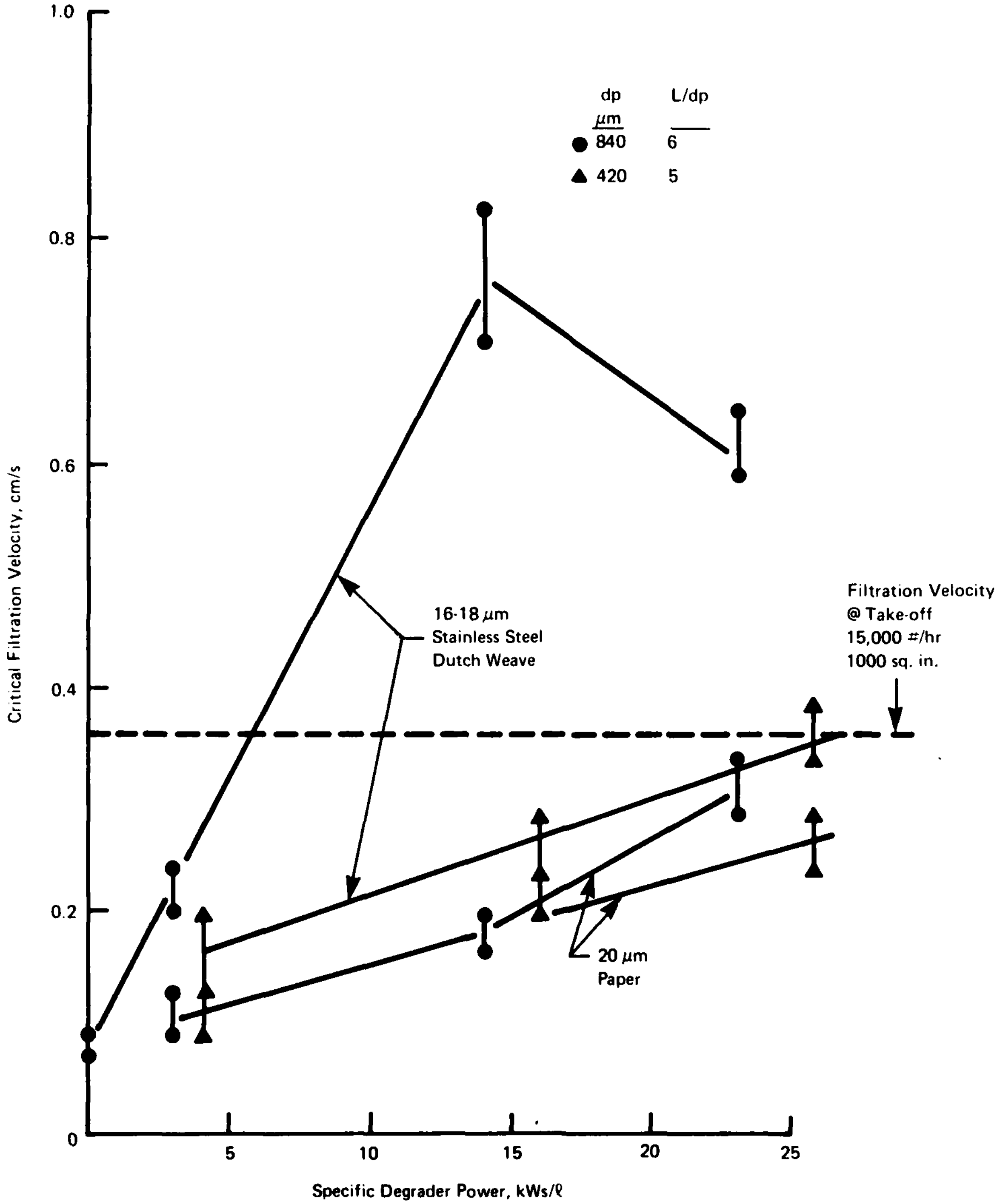

FIGURE 30. EFFECT OF SPECIFIC DEGRADER POWER, BEAD SIZE, AND FILTER MATERIAL ON THE CRITICAL FILTRATION VELOCITY (25°C)

TABLE 1. STEAM JET GUM* ANALYSIS OF AMK SAMPLES FROM FAA TECHNICAL CENTER (FUEL SPILLAGE TESTS)

FAA Technical Center Test Number	Wt% FM-9 Nominal	Wt% FM-9 Gum**
1	0.40	0.358
2	0.40	0.351
3	0.40	0.360
4	0.40	0.357
5	0.40	0.355
6	0.40	0.360
9	0.40	0.397
10	0.267	0.270
11	0.267	0.262
12	0.267	0.264
13	0.333	0.328
14	0.333	0.331
15A	0.333	0.346
22	0.30	0.303
23	0.30	0.304
24	0.30	0.292
25	0.30	0.306
26	0.30	0.304
27	0.30	0.300
30	0.30	0.301
31	0.30	0.295
33	0.20	0.195
34	0.20	0.194
36	0.20	0.194
38	0.20	0.193
41-44	0.30	0.296
45	0.30	0.302
49-53	0.250	0.228
55	0.30	0.287
59-61	0.26	0.265
62-64	0.26	0.267

* ASTM D-381

** Average of duplicate samples

IV. CONCLUSIONS

1. AMK can be degraded by high velocity flow through metal screens or packed tubes without significant plugging due to gel formation.

2. The degree of polymer degradation as measured by the filtration ratio increases with increasing power and at 15 kWs/l the filtration ratio is close to 1. This is equivalent to 30 HP at a fuel-flow rate of 10,000 lb/hr.

3. Using the filtration ratio as the criterion of degradation, degrader efficiency:

    a. is independent of tube length for $2 \leq L/dp \leq 6$.

    b. decreases with increasing tube length for $L/dp \geq 12$.

    c. is higher for smaller beads or screens.

4. The spinning disc test characterizes the mist flammability of AMK in terms of a critical velocity below which flame radiation is negligible compared to Jet A. This critical velocity appears to correspond to the pass/marginal air velocity in large-scale fuel spillage tests.

5. Using the critical disc velocity as the primary criterion of fire protection, it appears that:

    a. the glycol/amine carrier fluid increases the effectiveness of FM-9 in Jet A.

    b. there is no significant difference in the mist flammability of AMK with or without the amine component of the carrier fluid.

    c. the addition of hydrogen bonding agents that eliminate the gel-forming tendency of AMK does not adversely effect mist flammability.

    d. over the range of 20°-40°C (flash point = 51°C), fuel temperature has little effect on mist flammability.

    e. at a specific degrader power of 15 kWs/l and higher, standard AMK has mist flammability characteristics similar to Jet A.

    f. AMK without carrier fluid requires more power to degrade than standard AMK.

    g. AMK without amine requires less power to degrade than standard AMK.

5\. The laminar flow of AMK through filter media is characterized by a critical velocity below which flow resistance is determined by the low shear viscosity. At a slightly higher velocity, gel formation results in filter plugging in which the pressure drop across the filter increases with time.

6. Preliminary results indicate that the critical filtration velocity is a function of the degree of polymer degradation and filter properties, such as pore size and pore geometry.

7. The addition of a hydrogen-bonding agent to AMK increases the critical velocity, and at a concentration of 0.5 wt% it appears possible to filter undegraded AMK through nominal 20-micrometer paper with only a slight increase in the area of existing aircraft fuel filters.

8. For undegraded or lightly degraded AMK, there is a close correspondence between the critical filtration velocity and the average filtration velocity in the standard filtration test. However, for highly degraded AMK, the flow time in the standard test is too short to detect gel formation with the 16- to 18-micrometer metal screen. Consequently, the standard filtration ratio test overestimates the degree of degradation.

9. A pump filtration test that measures the pressure drop across a filter as a function of the velocity and flow times is a better test for intentional polymer degradation than the standard filtration test.

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